Console Acceptance Tests & Specifications

UNITY*INOVA NMR Spectrometer Systems*

Pub. No. 01-999120-00, Rev. B0800

VARIAN

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Table of Contents

SAFETY PRECAUTIONS 7

Chapter 1. Installation Tests and Demonstrations 11

1.1 Acceptance Testing 11
1.2 Computer Audit 11
1.3 Installation Checklist 12
1.4 System Documentation Review 12
1.5 Basic System Demonstration 12
Magnet Demonstration 12
Console and Probe Demonstration 12
1.6 General Acceptance Testing Requirements 13
General Requirements 13
Probe Installation Policies 14
1.7 AutoTest Automated Instrument Testing 15
Test Design 15
Scope of Test 15
Test Output 16
AutoTest Directory Structure 16
AutoTest Macros 19
Standard Tests Performed by AutoTest 21
Details of AutoTest Experiments 24
1.8 Varian Sales Offices 34
1.9 Posting Requirements for Magnetic Field Warning Signs 35
Warning Signs 35

Chapter 2. Console and Magnet Test Procedures 37

2.1 Automated Test Procedures—Running AutoTest 37
Installing Autotest 37
Sample for AutoTest 38
Setting Up for AutoTest 38
Running AutoTest 38
Saving Data and FID Files from Previous Runs 39
Creating Probe-Specific Files 40
Tests Performed by AutoTest 40
2.2 Manual Test Procedures Required to Demonstrate Console Operation 42
Homonuclear Decoupling 43
Lock Frequency Stability 43
Basic Variable Temperature Operation 44
Magnet Drift 45

WALTZ ^{1}H Decoupling—Preprogrammed Phase Modulation 46
WALTZ ^{1}H Decoupling—High-Performance RF Waveform Generator 47
2.3 Procedures for Contracted Custom Console Specifications 48
Temperature Accuracy for VT Systems 48
Stability Calibration for High-Stability VT Accessory 50
Homospoil Demonstration 51
Sucrose Anomeric ^{1}H Signal-to-Noise Ratio 52
Aqueous Phenylalanine Water Suppression 54

Chapter 3. Consoles and Magnets Specifications 57

3.1 Specifications for AutoTest 57
3.2 Specifications for Manual Console Tests 60
Lock Frequency Stability 61
Homonuclear Decoupling 62
Variable Temperature Operation 63
Magnet Drift Specifications 64
WALTZ ^{1}H Decoupling Using Preprogrammed Phase Modulation 65
WALTZ ^{1}H Decoupling Using High-Performance Waveform Generators . 66
3.3 Contracted Custom Console Specifications 67
Temperature Accuracy for VT Accessories 68
Stability Calibration for High-Stability VT Accessory 69
Homospoil Demonstration 70

Chapter 4. Acceptance Test Results 71

4.1 Computer Audit 73
4.2 System Installation Checklist 75
4.3 Supercon Shim Values 77
4.4 Console and Magnet Test Results 79
4.5 Consoles and Magnets Custom Specifications Form 81
Custom Specification 81
Sample Requirements 81
Name of Procedure Required for Custom Specification: 81

Index 83

List of Figures

Figure 1. 10-Gauss Warning Sign 36
Figure 2. 5-Gauss Warning Sign 36
Figure 3. Magnet Area Danger Sign 36
Figure 4. AutoTest Program 39

SAFETY PRECAUTIONS

The following warning and caution notices illustrate the style used in Varian manuals for safety precaution notices and explain when each type is used:

WARNING: ***Warnings*** **are used when failure to observe instructions or precautions could result in injury or death to humans or animals, or significant property damage.**

CAUTION: ***Cautions*** **are used when failure to observe instructions could result in serious damage to equipment or loss of data.**

Warning Notices

Observe the following precautions during installation, operation, maintenance, and repair of the instrument. Failure to comply with these warnings, or with specific warnings elsewhere in Varian manuals, violates safety standards of design, manufacturing, and intended use of the instrument. Varian assumes no liability for customer failure to comply with these precautions.

WARNING: **Persons with implanted or attached medical devices such as pacemakers and prosthetic parts must remain outside the 5-gauss perimeter from the centerline of the magnet.**

The superconducting magnet system generates strong magnetic fields that can affect operation of some cardiac pacemakers or harm implanted or attached devices such as prosthetic parts and metal blood vessel clips and clamps.

Pacemaker wearers should consult the user manual provided by the pacemaker manufacturer or contact the pacemaker manufacturer to determine the effect on a specific pacemaker. Pacemaker wearers should also always notify their physician and discuss the health risks of being in proximity to magnetic fields. Wearers of metal prosthetics and implants should contact their physician to determine if a danger exists.

Refer to the manuals supplied with the magnet for the size of a typical 5-gauss stray field. This gauss level should be checked after the magnet is installed.

WARNING: **Keep metal objects outside the 10-gauss perimeter from the centerline of the magnet.**

The strong magnetic field surrounding the magnet attracts objects containing steel, iron, or other ferromagnetic materials, which includes most ordinary tools, electronic equipment, compressed gas cylinders, steel chairs, and steel carts. Unless restrained, such objects can suddenly fly towards the magnet, causing possible personal injury and extensive damage to the probe, dewar, and superconducting solenoid. The greater the mass of the object, the more the magnet attracts the object.

Only nonferromagnetic materials—plastics, aluminum, wood, nonmagnetic stainless steel, etc.—should be used in the area around the magnet. If an object is stuck to the magnet surface and cannot easily be removed by hand, contact Varian service for assistance.

Warning Notices (*continued*)

Refer to the manuals supplied with the magnet for the size of a typical 10-gauss stray field. This gauss level should be checked after the magnet is installed.

WARNING: **Only qualified maintenance personnel shall remove equipment covers or make internal adjustments.**

Dangerous high voltages that can kill or injure exist inside the instrument. Before working inside a cabinet, turn off the main system power switch located on the back of the console, then disconnect the ac power cord.

WARNING: **Do not substitute parts or modify the instrument.**

Any unauthorized modification could injure personnel or damage equipment and potentially terminate the warranty agreements and/or service contract. Written authorization approved by a Varian, Inc. product manager is required to implement any changes to the hardware of a Varian NMR spectrometer. Maintain safety features by referring system service to a Varian service office.

WARNING: **Do not operate in the presence of flammable gases or fumes.**

Operation with flammable gases or fumes present creates the risk of injury or death from toxic fumes, explosion, or fire.

WARNING: **Leave area immediately in the event of a magnet quench.**

If the magnet dewar should quench (sudden appearance of gasses from the top of the dewar), leave the area immediately. Sudden release of helium or nitrogen gases can rapidly displace oxygen in an enclosed space creating a possibility of asphyxiation. Do not return until the oxygen level returns to normal.

WARNING: **Avoid liquid helium or nitrogen contact with any part of the body.**

In contact with the body, liquid helium and nitrogen can cause an injury similar to a burn. Never place your head over the helium and nitrogen exit tubes on top of the magnet. If liquid helium or nitrogen contacts the body, seek immediate medical attention, especially if the skin is blistered or the eyes are affected.

WARNING: **Do not look down the upper barrel.**

Unless the probe is removed from the magnet, never look down the upper barrel. You could be injured by the sample tube as it ejects pneumatically from the probe.

WARNING: **Do not exceed the boiling or freezing point of a sample during variable temperature experiments.**

A sample tube subjected to a change in temperature can build up excessive pressure, which can break the sample tube glass and cause injury by flying glass and toxic materials. To avoid this hazard, establish the freezing and boiling point of a sample before doing a variable temperature experiment.

Warning Notices (*continued*)

WARNING: **Support the magnet and prevent it from tipping over.**

The magnet dewar has a high center of gravity and could tip over in an earthquake or after being struck by a large object, injuring personnel and causing sudden, dangerous release of nitrogen and helium gasses from the dewar. Therefore, the magnet must be supported by at least one of two methods: with ropes suspended from the ceiling or with the antivibration legs bolted to the floor. Refer to the *Installation Planning Manual* for details.

WARNING: **Do not remove the relief valves on the vent tubes.**

The relief valves prevent air from entering the nitrogen and helium vent tubes. Air that enters the magnet contains moisture that can freeze, causing blockage of the vent tubes and possibly extensive damage to the magnet. It could also cause a sudden dangerous release of nitrogen and helium gases from the dewar. Except when transferring nitrogen or helium, be certain that the relief valves are secured on the vent tubes.

WARNING: **On magnets with removable quench tubes, keep the tubes in place except during helium servicing.**

On Varian 200- and 300-MHz 54-mm magnets only, the dewar includes removable helium vent tubes. If the magnet dewar should quench (sudden appearance of gases from the top of the dewar) and the vent tubes are not in place, the helium gas would be partially vented sideways, possibly injuring the skin and eyes of personnel beside the magnet. During helium servicing, when the tubes must be removed, carefully follow the instructions and safety precautions given in the manual supplied with the magnet.

Caution Notices

Observe the following precautions during installation, operation, maintenance, and repair of the instrument. Failure to comply with these cautions, or with specific cautions elsewhere in Varian manuals, violates safety standards of design, manufacturing, and intended use of the instrument. Varian assumes no liability for customer failure to comply with these precautions.

CAUTION: **Keep magnetic media, ATM and credit cards, and watches outside the 5-gauss perimeter from the centerline of the magnet.**

The strong magnetic field surrounding a superconducting magnet can erase magnetic media such as floppy disks and tapes. The field can also damage the strip of magnetic media found on credit cards, automatic teller machine (ATM) cards, and similar plastic cards. Many wrist and pocket watches are also susceptible to damage from intense magnetism.

Refer to the manuals supplied with the magnet for the size of a typical 5-gauss stray field. This gauss level should be checked after the magnet is installed.

Caution Notices (*continued*)

CAUTION: **Keep the PCs, (including the LC STAR workstation) beyond the 5-gauss perimeter of the magnet.**

Avoid equipment damage or data loss by keeping PCs (including the LC workstation PC) well away from the magnet. Generally, keep the PC beyond the 5-gauss perimeter of the magnet. Refer to the *Installation Planning Guide* for magnet field plots.

CAUTION: **Check helium and nitrogen gas flowmeters daily.**

Record the readings to establish the operating level. The readings will vary somewhat because of changes in barometric pressure from weather fronts. If the readings for either gas should change abruptly, contact qualified maintenance personnel. Failure to correct the cause of abnormal readings could result in extensive equipment damage.

CAUTION: **Never operate solids high-power amplifiers with liquids probes.**

On systems with solids high-power amplifiers, never operate the amplifiers with a liquids probe. The high power available from these amplifiers will destroy liquids probes. Use the appropriate high-power probe with the high-power amplifier.

CAUTION: **Take electrostatic discharge (ESD) precautions to avoid damage to sensitive electronic components.**

Wear a grounded antistatic wristband or equivalent before touching any parts inside the doors and covers of the spectrometer system. Also, take ESD precautions when working near the exposed cable connectors on the back of the console.

Radio-Frequency Emission Regulations

The covers on the instrument form a barrier to radio-frequency (rf) energy. Removing any of the covers or modifying the instrument may lead to increased susceptibility to rf interference within the instrument and may increase the rf energy transmitted by the instrument in violation of regulations covering rf emissions. It is the operator's responsibility to maintain the instrument in a condition that does not violate rf emission requirements.

Chapter 1. Installation Tests and Demonstrations

Sections in this chapter:

- 1.1 “Acceptance Testing” this page
- 1.2 “Computer Audit” this page
- 1.3 “Installation Checklist” page 12
- 1.4 “System Documentation Review” page 12
- 1.5 “Basic System Demonstration” page 12
- 1.6 “General Acceptance Testing Requirements” page 13
- 1.7 “AutoTest Automated Instrument Testing” page 15
- 1.8 “Varian Sales Offices” page 34
- 1.9 “Posting Requirements for Magnetic Field Warning Signs” page 35

Following each installation of a Varian UNITY*INOVA* NMR spectrometer system, an installation engineer tests and demonstrates the instrument’s operation. Chapter 4, “Acceptance Test Results,” outlines the procedures for the acceptance tests. The forms for recording test results in that chapter follow the same sequence as the tests.

1.1 Acceptance Testing

The objectives of the acceptance tests procedures are twofold:

- To identify the tests to be performed during system installation.
- To identify the precise methods by which these tests are performed.

The procedures are ordered in a sequence designed to efficiently and logically evaluate the performance of the instrument. These procedures cover the basic specifications of the instrument, that is, signal-to-noise (S/N), resolution, and lineshape, and are not intended to reflect the full range of operating capabilities or features of a research NMR spectrometer.

Note: Performance of any additional tests beyond those described herein must be agreed upon in writing as part of the customer contract. Test samples for these contracted tests are not supplied by Varian.

1.2 Computer Audit

“Computer Audit,” page 73, includes a computer audit form. The information from this form will help Varian personnel assist you better in making future software upgrades and avoiding hardware compatibility problems. You will be asked for information about all computers directly connected to the spectrometer or used to process NMR data.

1.3 Installation Checklist

"System Installation Checklist," page 75, includes an installation checklist form.

1.4 System Documentation Review

Following the completion of the acceptance tests and computer audit, the following system documentation will be reviewed with the customer:

- Software Object Code License Agreement
- Varian and OEM manuals
- Warranty coverage and where to telephone for information

1.5 Basic System Demonstration

The basic operation of the system is demonstrated to the primary user. The objective of the demonstration is to familiarize the customer with system features and safety requirements, as well as to assure that all mechanical and electrical functions are operating properly. Detailed specifications and circuit descriptions will not be covered.

Note: Varian installation engineers are not responsible for nor trained to run any spectra not described in the acceptance tests procedures.

Magnet Demonstration

The magnet demonstration includes the following items:

- Posting requirements for magnetic field warning signs
- Cryogenics handling procedures and safety precautions
- Magnet refilling
- Flowmeters
- Homogeneity disturbances

Console and Probe Demonstration

The console and probe demonstration includes the following items:

- Loading programs and operating the streaming magnetic tape unit.
- Experiment setup, including mounting the probe in the magnet.
- Basic instrument operation to obtain typical spectra, including probe tuning, magnet homogeneity shimming, and printer/plotter operation.
- Demonstration of broadband operation.
- Demonstration of homonuclear and heteronuclear decoupling.

Formal training in the operation and maintenance of the spectrometer is conducted by Varian NMR Systems at periodically scheduled training seminars held in most Varian Application Laboratories. On-site training is available in some geographic locations. Contact your sales representative for further information on availability and pricing for these courses.

To make the system demonstration most beneficial, the customer should review Varian and OEM manuals before viewing the demonstration.

1.6 General Acceptance Testing Requirements

Each Varian UNITY*INOVA* spectrometer is designed to provide high-resolution performance when operated in an environment as specified in the UNITY*INOVA NMR Spectrometer Systems Installation Planning Guide*. Unless both the specific requirements of this manual and the general requirements specified in the UNITY*INOVA Installation Planning Guide* are met, Varian cannot warrant that the NMR spectrometer system will meet the published specifications.

General Requirements

- The UNITY*INOVA* performance specifications in effect at the time the system is ordered are used to evaluate the system.
- The appropriate quarter-wavelength cable must be used for each nucleus.
- Homogeneity settings must be optimized for each sample (manual shimming may be required in any or all cases). The shim parameters for resolution tests on each probe should be recorded in a log book and in a separate file (in the `/vnmr/shims` directory) for each probe. For example, for a 5-mm switchable probe, the shim parameters can be saved with the command `svs('/vnmr/shims/sw5res')`. These values can then be used as a starting point when adjusting the homogeneity on unknown samples, by using `rts('sw5res')`.
- The probe must be tuned to the appropriate frequency.
- The spinning speed used is the following:

Sample (mm)	*Nuclei*	*Speed (Hz)*
5	all	20–26
10	all	15

Note: Making 10-mm tubes spin faster than 15 Hz may cause vortexing in samples, severely degrading the resolution.

- All test parameters are stored in the disk library `/vnmr/tests` and can be recalled by entering `rtp('/vnmr/tests/`*xxx*`')`, where *xxx* is the name of the test, for example, `rtp('/vnmr/tests/H1sn')`. To see the parameter sets available for the standard tests, enter `ls('/vnmr/tests')`.
  - For all sensitivity tests, the value of `pw` must be changed to the value of the 90° pulse found in the pulse width test on the same probe.
  - For all direct observe pulse width tests, an appropriate array of `pw` values must be entered to determine the 180° pulse. The 180° pulse is the first non-zero pulse that gives minimum intensity of the spectrum. The 180° pulse is usually determined by interpolation between a value that gives a positive signal, and a value that gives a negative signal. The 90° pulse width is one half the 180° pulse.
- Signal-to-noise (S/N) is measured by the computer as follows:

$$\text{S/N} = \frac{\text{maximum amplitude of peak}}{2 \times \text{root mean square of noise region}}$$

- Lineshape should be measured with the aid of the system software. The properly scaled spectra should also be plotted and retained.
- Software determination of lineshape:
    a. Display and expand the desired peak.
    b. Enter **nm**, then **dc** for drift correction to ensure a flat baseline. Set **vs=10000**. Press **F7** until **Thresh** is displayed on axis f2. Press the **F2** key to display the horizontal threshold cursor. Set **th=55** (the 0.55% level).
    c. Press the **F1** key to display two vertical cursors, and align them on the intersections of the horizontal cursor and the peak. Enter **delta?** to see the difference, in Hz, between the cursors.
    d. Set **th=11** (the 0.11% level) and repeat the first three steps.
- Determination of lineshape from a plot:
    a. Use a large enough plot width to allow accurate determination of the baseline. The baseline should be drawn through the center of the noise, in a region of the spectrum with no peaks.
    b. The 0.55% and 0.11% levels are then measured from the baseline and calculated from the height of the peak and the value of vs. For example, if a peak is 9.0 cm high with vs=200, the 0.55% level on a 100-fold vertical expansion (vs=20000) is 9.0×0.55, or 4.95 cm from the baseline.

  If the noise is significant at the 0.55% and 0.11% levels, the linewidth should be measured horizontally to the center of the noise.
- For all sensitivity tests, a noise region free of any anomalous features should be chosen with the cursors. Neither cursor should be placed any closer to an edge of the spectrum than 10 percent of the value of sw. This should produce the best possible signal-to-noise that is representative of the spectrum.
- The results of all tests should be plotted as a permanent record. Include a descriptive label and a list of parameters. These plots can then be saved as part of the acceptance tests documentation.

Probe Installation Policies

The following policies are in effect at installation:

- *Custom Specifications Policy* – For custom specifications that have been purchased, record test results in "Consoles and Magnets Custom Specifications Form," page 81.
- *Specifications Policy for Probes Used in Systems other than* UNITY*INOVA* – For probes purchased for use in systems other than Varian UNITY*INOVA* systems, no guarantee is given that these probes meet current specifications.
- *Testing Policy for Indirect Detection Probes for Direct Observe Broadband Performance* – Probes designed for indirect detection applications are tested for indirect detection performance only.
- *Sample Tubes Policy* – Tests are performed with the following sample tubes:

  3-mm probes: 3-mm tubes with 0.30-mm wall (Wilmad 327-PP, or equivalent).

  5-mm probes: 5-mm tubes with 0.38-mm wall (Wilmad 528-PP, or equivalent).

  8-mm probes: 8-mm tubes with 0.51-mm wall (Wilmad 513A-7PP, or equivalent).

  10-mm probes: 10-mm tubes with 0.46-mm wall (Wilmad 513-7PP, or equivalent).

  Using sample tubes with thinner wall thickness (e.g., Wilmad 5-mm 545-PPT, or equivalent; Wilmad 10-mm 513-7PPT, or equivalent) increases signal-to-noise.

1.7 AutoTest Automated Instrument Testing

AutoTest is a test suite designed as an instrument performance tracking tool for UNITY*INOVA* NMR spectrometers. "Automated Test Procedures—Running AutoTest," page 37, describes how to install AutoTest and how to use it for automated instrument testing.

After AutoTest is installed, the user can establish benchmark values for a spectrometer and use these values to track the system's performance characteristics at regular intervals. The test is completely automated and can be performed in about 40 to 90 minutes, depending on the options chosen.

Significant deviations from the norm can provide an indication of a hardware failure or slow degradation, each of which might be hard to identify using normal spectra. Tests have been designed to test only one aspect of performance and one piece of hardware at a time, as much as possible. Historical trends in performance can be displayed.

This section contains the following topics:

- "Test Design," this page
- "Scope of Test," this page
- "Test Output," page 16
- "AutoTest Directory Structure," page 16
- "AutoTest Macros," page 19
- "Standard Tests Performed by AutoTest," page 21
- "Details of AutoTest Experiments," page 24

Test Design

The test is designed to quantitate instrument performance on an unbiased statistical basis. The average values and standard deviations from stability and reproducibility measurements are determined. Other tests produce a regular intensity output (exponential or linear, such as when an attenuator or modulator value is varied). Correlation coefficients and standard deviations from a regression analysis are reported.

Calibrations are made of parameters such as ^{1}H `pw90` using channels 1 and 2, ^{13}C `pw90`, and high- and low-band amplifier compressions. Probe rf homogeneity (^{1}H 450/90, 810/90, and ^{13}C 360/0, 720/0) values are determined. Sensitivity, T_1, and T_2 are measured.

Scope of Test

Various hardware aspects are checked, including:

- Lock performance (measured by phase-cycle cancellation efficiency)
- Image cancellation
- Built-in phase modulator decoupling (MLEV-16, WALTZ-16, XY-32, and GARP-1)
- Waveform generation (WFG) based decoupling (WURST and STUD)
- Heating under ^{13}C decoupling and ^{1}H spinlocks
- Variable temperature response

Functionality of receiver gain, small-angle phase shifters, pulse turn-on, attenuators, modulators, and pulse shaping is measured. The lock channel power and gain control is checked and quantified.

Gradient performance is checked on all active axes specified by the pfgon parameter. The tests include signal amplitude stability following a pulse done 100 µs after a gradient pulse, or a pulse followed by a bipolar gradient pair generating a gradient echo. Field recovery is measured by doing a gradient followed by a variable delay and an rf pulse. Gradient DAC values to generate 10 G/cm strengths are determined. Gradient stability is measured by measuring a CPMGT2 experiment with and without gradients within the spin echoes.

Test Output

AutoTest results are plotted (if specified), FIDs are stored, and results are logged in appropriate text files. At the end of the test a single-page report is printed, summarizing the test results. If desired, the test can be made to repeat until aborted to acquire multiple runs.

After several runs have been completed, histograms showing all previous values of a parameter can be viewed, for example, all previous z-axis gradient calibrations, along with the average of all the results and their standard deviations. Of particular interest is any parameter exhibiting a sudden change in value or a steady increase or decrease. These results can give an early indication for a hardware problem.

AutoTest Directory Structure

AutoTest uses the `~/vnmrsys/autotest` directories listed in Table 1.

Table 1. AutoTest Directories.

Directory	*Contents*
`data`	FIDs from the recent AutoTest run(s)
`data.failed`	FIDs from any failed Auto Test experiments
`history`	History files for the various tests
`reports`	Copies of the report generated each time AutoTest is run
`parameters`	Parameter files—default entry is `standard.par`
`texts`	Copies of the text files attached to the AutoTest experiments
`atdb`	AutoTest database

data Directory

The `~/vnmrsys/autotest/data` directory contains FIDs collected in previous AutoTest experiments. As each experiment finishes, the macro specified by the `wexp` parameter executes, and as part of that macro, a `svf` command is performed that saves the FID under a file name specified by the parameter `at_currenttest` (if it contains a name). The macro first removes any file by the same name (the results of the test from the last time it was run) and then executes `svf`. Thus, the data directory may contain FIDs obtained during different AutoTest runs if those runs were not full runs.

Any data files stored in the data directory can be recalled by normal VNMR commands such as `rt`. The data may then be transformed and displayed. The `wexp` parameter will contain the name of the macro normally used for data processing, so that the `wexp` command can be used to duplicate the actions normally done in an automatic manner.

CAUTION: **If file `~/vnmrsys/autotest/atdb/at_selected_tests` is empty, only processing and no further acquisition is done.**

This result is normally the case if the last AutoTest run came to a normal completion. However, if the last AutoTest run was aborted and no new entry into the AutoTest Program was done, this file will contain entries and an acquisition may start up following the `wexp` command. If so, just abort the acquisition.

data.failed Directory

The `~/vnmrsys/autotest/data.failed` directory contains any data from any failed experiment. Failure results when a calculated result falls outside limits defined in the `~/vnmrsys/autotest/atdb/at_spec_table` file. Varian specifications are indicated in the `~/vnmrsys/autotest/atdb/at_spec_table` file. Users can modify this file by supplying upper and lower limits. Any user-modified `at_spec_table` file should be saved outside `~/vnmrsys/autotest`, since this file can be deleted later.

parameters Directory

The `~/vnmrsys/autotest/parameters` directory contains any parameter set used by AutoTest macros, including any put there by the user. Normally, only `standard.par` is present. This parameter set has all parameters necessary for the AutoTest macros. Values of parameters may be displayed by using `dg` in the text window. Some parameters are only displayed when certain variables are nonzero, or `'y'` if a string parameter; however, these parameters are printed and displayed if used in an experiment. The AutoTest macro `Atrtp` is used to recall a parameter set from this directory.

reports Directory

The `~/vnmrsys/autotest/reports` directory contains text files from previous AutoTest runs, by date. Each run produces a report, whether plotting is requested or not. The report file for a currently proceeding AutoTest run is `~/vnmrsys/autotest/REPORT`. At the end of an AutoTest run, this file is copied to the reports directory under a title that includes the date and time. If AutoTest is repeated, automatically a new report is written out for each complete AutoTest run. The existing `~/vnmrsys/autotest/REPORT` file is renamed as `~/vnmrsys/autotest/LASTREPORT` whenever an AutoTest run begins. Similar actions are executed for the `atrecord_report`.

texts Directory

The `~/vnmrsys/autotest/texts` directory contains mainly text files that are printed on some spectral plots and most parameter set printouts. These files explain the purpose of the test.

history Directory

The `~/vnmrsys/autotest/history` directory contains text files that record the values determined in AutoTest runs. They are generated automatically by the `ATrecord` macro which is used in any AutoTest macro that obtains a numerical result from an NMR experiment. Each result is written on a new line and is date-stamped. Tests that have a Varian specification listed in this manual will be denoted as having passed or failed.

If the `history` file has more than one result per line, any one failure will cause a fail result for the whole line. When the `history` file is viewed using the History display (after using the macro `autotest`), failure is indicated by a red data point in graphical output and a colored entry in the text output.

atdb Directory

The `~/vnmrsys/autotest/atdb` directory contains mainly the following text files used by the Auto Test program to create the AutoTest interface:

at_tests_file

The `at_tests_file` file defines all the tests that AutoTest can perform. Tests are specified by a macro name and description. Normally, these are grouped and separated by a line starting with `Label`. The word following will be displayed as a heading for a group of tests. The test descriptions are displayed in the Test Library display (after entering the macro `autotest` or by using a menu calling this macro). The macro names are not shown in the display; checkboxes are displayed next to the test description.

New tests may be added to the `at_tests_file` by specifying a group title (use the `Label` keyword as the first word on the line, followed by a descriptive phrase). Specify a macro name and then a test description, one per line.

at_groups_file

The `at_groups_file` file defines test `packages` that have been assembled for convenience. Each package has a line that gives a description (in double quotes) followed by a list of macros to be used in the order of acquisition. There are no restrictions on the placement of these macros in the text file, only the order matters. When the next double-quoted entry appears, a new group is set.

The AutoTest interface display shows these packages as checkbox entries in the Configuration display. Selection of one or more of these causes their execution in the order of selection, once the Begin Test button is selected. When this happens, the `at_selected_tests file` is fixed. Selection of the All Tests checkbox disables all the other selections because they will be done as part of the All Tests run.

Users may add new packages to the Configuration display list by adding appropriate lines to the `at_groups_file` in the same format.

at_selected_tests

The `at_selected_tests` file contains the names of the macros to be run as part of the AutoTest procedure and is fixed at the time the Begin Test button is selected. The format is one line per macro with each line containing the name of a macro, in the order of acquisition.

As AutoTest proceeds, each line is deleted as the specified macro finishes its activity. Thus, completion of the AutoTest run is defined as when this file is empty. The single exception is the case of automatic repeating of AutoTest, as specified by the Repeat Until Stopped checkbox in the Configuration display and as indicated by the value of the global parameter `at_cycletest('y')`. In this case, at the completion of the AutoTest run, the contents of the file `at_cycled_tests` are copied into `at_selected_tests` and the process then continues.

at_cycled_tests

The `at_cycled_tests` file is updated when the Begin Test button is selected. If the Repeat Until Stopped checkbox is selected in the Configuration display, the global parameter `at_cycletests` is set to `'y'` and the file `at_selected_tests` is copied to `at_cycled_tests`. If no test cycling is requested, this file is emptied.

at_spec_table

The `at_spec_table` file is written out when the `~/vnmrsys/autotest` directory is created and is spectrometer-dependent. The appropriate file is copied from the directory `/vnmr/autotest`, depending on spectrometer frequency. It contains a list of macros used in AutoTest. For each macro the following is specified:

- The history file affected by the macro.
- The column number (not counting date) containing the result.
- The lower limit for the result.
- The upper limit for the result.
- A text description of the result. This text description is used for the graphical displays and plots. A comment line above each macro serves to describe the test.

All results specified in this manual have upper and lower limits specified numerically in this file. Those not having Varian specifications have asterisks (*) as entries for upper and lower limits and these results will have no indication of pass or fail in their history files, or colored indication of failure in the graphical displays of the history files.

Users may wish to set their own upper and lower limits for many, if not all, of the results. They may do so by replacing the asterisks with numbers. Of course, this should only be done after a good statistical base is obtained, such as more than 20 complete AutoTest runs. Once this base is obtained, the numbers put into the `at_spec_table file` should have a reasonable margin of error built in.

It is a good idea to make a copy of the file `at_specs_table file` prior to changing it, as well as the modified file, because deletion or renaming of the `autotest` directory will result in a default `at_spec_table` being copied from `/vnmr/autotest/atdb`.

AutoTest Macros

To help users who may want to add tests or modify tests, this section describes some of the macros used by AutoTest. These macros are in `/vnmr/maclib/maclib.autotest`.

ATglobal Macro

The `ATglobal` macro is run when the AutoTest program begins. The macro checks for the existence of `autotest` parameters in the user file `~/vnmrsys/global`. These parameters are used to store calibrations and results that are used by `autotest` macros. If the parameters are not present, `ATglobal` creates them. Otherwise, the parameters are left unchanged. A partial list of these parameters is given in Table 2.

ATstart Macro

The `ATstart` macro is run after the Begin Test button is selected in the Configuration display. The macro sets the global parameters to reflect the current state of the hardware and aborts under certain circumstances, such as if requested tests are not compatible with the current hardware settings.

Messages are displayed indicating the source of the problem. The reports are initialized with relevant information and the ATnext macro is executed.

ATnext Macro

The `ATnext` macro checks the `at_selected_tests` file and copies the first entry into the global parameter `at_cur_smacro`, deletes the top line in `at_selected_tests`

Table 2. Selected Parameters Created by ATglobal.

Parameter	*Contains*
`at_currenttest`	Name under which the FID is stored
`autotestdir`	Full path of the `autotest` directory
`at_user`	Name of the user running `autotest` (printed in report)
`at_coilsize`	length (in mm) of active window in coil (typically 16 or 18 mm)
`at_consoletype`	Name of console entered in AutoTest window
`at_consolesn`	Number of console entered in AutoTest window
`at_probetype`	Name entered for probe used in AutoTest window
`at_wntproc`	y or n (for processing/display after each FID)
`at_cycletest`	y or n (for automatic repeating of AutoTest)
`at_printparams`	y or n (for parameter list/pulse sequence printouts)
`at_plotauto`	y or n (for automatic plotting)
`at_graphhist`	y or n (for history graphs plotting)
`at_locktests`	y or n (for lock power/gain tests)
`at_T1`	Value of last determined T_1
`at_gain`	Value of gain determined by autogain
`at_tof`	Value of `tof` for water
`at_fsq`	Value of `fsq` parameter
`at_dsp`	Current value of `dsp` at start of run
`at_ampl_compr`	Value of high-band amplifier compression
`at_LBampl_compr`	Value of low-band amplifier compression
`at_decHeating`	Temperature increase from decoupling
`at_linewidth`	Linewidth of water resonance
`at_pw90`	90° `pw` at power specified in AutoTest display
`at_tpwr`	Power specified in AutoTest display
`at_pw90Lowpower`	90° `pw` at reduced power
`at_tpwrLowpower`	Power level at reduced power
`at_pw90_ch2`	90° `pw` on channel 2
`at_pwx90`	^{13}C `pw90` determined at `at_pwx90lvl`
`at_pwx90lvl`	^{13}C power level for approximately 15-µs pwx90
`at_pwx90Lowpower`	^{13}C `pw90` at reduced power
`at_pwx90Lowpowerlvl`	^{13}C power level at reduced power
`at_vttest`	y or n (for VT test)
`at_temp`	Current temperature
`at_vttype`	Current value of global parameter `vttype`
`at_tempcontrol`	Value reflects usage of `temp` `tcl/tk` panel
`at_gradtests`	y or n (for gradient tests)
`at_pfgon`	Current value of `pfgon`
`at_gmap`	y or n (for gradient mapping/shimming)
`at_gzcal`	Value of G/cm per DAC unit for z-axis gradient
`at_gxcal`	Value of G/cm per DAC unit for x-axis gradient
`at_gycal`	Value of G/cm per DAC unit for y-axis gradient

and executes the macro specified by `at_cur_smacro`. If the `at_selected_tests` file is empty, `ATnext` either finishes the AutoTest run or calls the `ATrestart` macro which copies the `at_cycled_tests` file to a`t_selected_tests`, permitting repeated AutoTest runs until manually aborted by the user.

`ATnext` is usually found at the bottom of each macro defining a particular test. This permits the linking of one test to another, in a general fashion.

ATxxx Macros

Specific tests usually have the designation of AT, followed by a number or group of letters. Each macro is self-contained, having the ability of setting up parameters, performing acquisition, processing the acquired data, possibly setting up new experiments and processing the data acquired from those experiments, creating plots, parameter printouts, archiving the raw data, performing statistical analyses of the data, and writing results to history files and reports.

A new MAGICAL capability is used that makes the macro easier to read and write, the `elseif` statement. This removes the need for multiple `endif` statements if multiple calls to the same macro are used.

To better illustrate the structure of these macros, Table 3 gives the source code for macro `AT16`, the turn-on test (channel 1). A column of descriptive comments has been added to clarify the statements.

The AutoTest macros can be run as independent macros if a specific test is desired. This is done by entering the macro in the VNMR command line. Again, if the file `at_selected_tests` is not empty, the `ATnext` macro will start a new acquisition.

Standard Tests Performed by AutoTest

Automated Console Tests

- 90° pulse stability channel 1 and channel 2
- 30° amplitude stability channel 1 and channel 2
- Pulse turnon time channel 1 and channel 2
- Phase cycle cancellation (2-scan test is run as demonstration, no specification is set)
- Quadrature image: 1 scan and 4 scans
- Frequency-shifted quadrature image: 1 scan
- Phase stability test (13° test) channel 1 and channel 2
- Attenuator test channel 1 and channel 2
  Full power correlation coefficient and standard deviation
  Reduced power correlation coefficient and standard deviation
- Modulator linearity channel 1 and channel 2
  `tpwr=40`: standard deviation
  `tpwr=-16`: standard deviation
- Temperature increase in spinlock test
- Lock power test correlation coefficient
- Lock gain test correlation coefficient
- Variable temperature test

Table 3. Source Code for AT16 Macro Example.

Code	*Comment*
`if ($#=0) then`	First time AT16 is run it has no arguments.
`ATrtp('standard')`	Recalls standard parameter set.
`text('Pulse Turnon Test')`	
`at_currenttest=turnon_ch1`	Puts name of test in global variable.
`tpwr=tpwr-6 ph`	
`array('pw',37,0.1,.025)`	Sets up pulse width array.
`ss=2`	
`wnt='ATwft select(celem) aph0 vsadj dssh dtext'`	Specifies what to do every FID
`wexp='AT16(`PART1`)'`	Specifies what to do at end of experiment.
`ATcycle`	Disables wnt processing if in repeat mode.
`au`	Begins acquisition and specifies wnt/wexp processing to occur.
`write('line3','Pulse Turnon Test (channel 1)')`	
`dps`	
`elseif ($1='PART1') then`	This part executes at end of experiment.
`if (at_plotauto='y') then`	
`if (at_printparams='y') then`	
`pap ATpltext`	If parameter printout requested.
`pps(120,0,wcmax-120,90)`	
`page`	
`endif`	
`endif`	
`select(arraydim) aph0`	
`f peak:$ht,cr rl(0) sp=-1p wp=2p vsadj dssh dtext`	
`ATreg6`	Fits to straight line and displays/plots data.
`ATpl3:$turnon,$corrcoef`	Determines turn-on time and correlation coefficients
`$turnon=trunc($turnon) $corrcoef= trunc(1000*$corrcoef)/1000`	Limits number of decimal places.
`ATrecord('TURNONch1','Pulse Turnon Time (nsec) (channel 1)','time  ',$turnon,' corr_coef.',$corrcoef)`	Writes out results to history file.
`write('file',autotestdir+'/REPORT', 'Pulse Turnon Time (channel 1): %2.0f nsec.-Corr. Coef. = %1.3f ',$turnon,$corrcoef)`	Writes results to report.
`if (at_plotauto='y') then`	
`ATpltext(100,wc2max-5)`	
`full wc=50 pexpl page`	Plots regression fit
`endif`	
`ATsvf`	Removes old data set and stores FID under name in at_currenttest.
`ATnext`	Starts next macro in at_selected_tests file, if present.
`endif`	Closes elseif part

Automated Tests with Shaped RF

- Gaussian 90° stability, channel 1 and channel 2
- Gaussian phase stability test, channel 1 and channel 2
- Gaussian SLP phase stability test, channel 1 and channel 2

Automated Decoupling Performance Tests

- ^{13}C phase modulation decoupling profiles
  ^{13}C GARP decoupling profile
  ^{13}C WALTZ-16 decoupling profile
  ^{13}C XY-32 decoupling profile
  ^{13}C MLEV-16 decoupling profile
- ^{13}C adiabatic decoupling profiles (if waveform generator present on decoupling channel):
  ^{13}C STUD decoupling profile
  ^{13}C WURST decoupling profile
- Sample heating during ^{13}C broadband decoupling

90° Pulse Width Calibrations (PW90)

- ^{1}H 90° pulse width calibrations on channels 1 and 2
- ^{13}C 90° pulse width calibrations (PW90)

RF Homogeneity Tests

- ^{1}H rf homogeneity test
- ^{13}C rf homogeneity test

Gradient Calibrations and Performance Tests

- Gradient level for 10 G/cm along the following:
  Z axis for all gradient probes
  X axis for triax probes
  Y axis for triax probes
- Gradient echo stability for the following:
  Z axis at 30 G/cm
  X axis at 10 G/cm
  Y axis at 10 G/cm
  Z axis at 10 G/cm
- Gradient recovery stability for the following:
  X axis at 10 G/cm
  Y axis at 10 G/cm
  Z axis at 10 G/cm
- Gradient recovery for the following:
  X axis at ± 10 G/cm
  Y axis at ± 10 G/cm
  Z axis at ± 10 G/cm
- Cancellation after gradient.

Console Demonstration Tests

- High-band amplifier compression
- 1 µsec pulse amplitude stability channel 1 and channel 2
- Low-band amplifier compression
- Temperature rise in decoupler heating test
- AutoGain result for 90° pulse.
- Receiver gain (normal sampling 10-kHz sweep width)
- Receiver gain (oversampling 100-kHz sweep width)
- Folded noise reduction with large spectral width
- Phase switch settling time
  CPMG T_2 result for the following:
  - Gradient level = 10 G/cm
  - Without gradients
  - 1% gradient mismatch

Shaped RF Demonstrations

- Shaped pulse accuracy—waveform generator gaussian profile
- RF amplitude predictability using a gaussian pulse
- Amplitude scaling of shaped pulses using a gaussian pulse
- RF excitation predictability using a variety of shaped pulses.

Details of AutoTest Experiments

This section provides descriptions of the experiments performed by AutoTest.

All units of the rf system (transmitters, linear modulators, rf attenuators, amplifiers, receivers, and probes) must be in the standard configuration when AutoTest is run. If the system configuration has been changed, it must be returned to the standard configuration before running AutoTest for acceptance testing.

All data is stored, and both plots and statistical analyses are provided as part of the acceptance testing. Plots and statistical analyses are made concurrently with acquisition.

RF Performance Test (Nonshaped Channels 1 and 2)

Pulse Stability

Experiment – A single-scan pulse experiment is repeated 20 times and the spectra plotted in a horizontal stack. The average peak amplitude and rms deviation are measured and reported. This test is run for the following:

- 90° flip pulses
- 30° flip pulses
- 1 µsec pulses

Purpose of 90° Pulse Stability – Modern experiments require very high pulse reproducibility to minimize cancellation residuals and T_1 noise in 2D experiments. This test checks amplitude reproducibility by comparing a series of spectra obtained with the signal

following a single 90° pulse. The statistical analysis produces an rms deviation, in percent of the average peak height.

Purpose of 30° Pulse Stability – The sinusoidal nature of the excitation profile makes the signal generated following a 90° pulse less sensitive to error than signal following a much smaller flip angle pulse (the top of a sine wave is broad and changes in amplitude less for small changes in flip angle than for a smaller pulse). A 0° flip angle would have the highest sensitivity to flip angle, but would give no signal, of course. A compromise between the extremes of large signal following a 90° pulse, and no signal following a 0° pulse is to use a 30° pulse. The rms deviation is measured from an array of spectra obtained using 30° pulses.

Purpose of 1 μsec Pulse Stability – This test emphasizes the turnon characteristics of the pulse. Any instability of the pulse rise should give a corresponding reduction of measured stability. Since the flip-angle is much less than a 90° or 30° pulse, the measured stability may be lower. The rms deviation is measured from an array of spectra obtained using 1 μsec pulses.

Cancellation Test

Experiment – Four single-scan, 4 two-scan, and 4 four-scan 90° pulse spectra are acquired in which the transmitter phase is held fixed and the receiver is phase-cycled 0, 2, 1, 3. Data are plotted in a horizontal stack with the single-scan spectra on scale. The vertical scale is increased by 100 times and plotted in the same manner. Average residual signal for 2-scan and 4-scan cancellation are determined.

Purpose – Modern experiments (HMQC, HSQC, NOE-difference, etc.) often rely on phase-cycling to achieve desired results. This test compares single-transient response versus two- and four-transient response in which the phase-cycling is set to achieve cancellation.

Phase Stability (13° Phase Error) Test

Experiment – The 90° pulse stability test is repeated but uses a 90° pulse–1 ms—90° pulse train with the carrier positioned 37 Hz off-resonance from the water.

Purpose – Phase stability is essential for high-performance modern experiments. Poor phase stability would produce poorer water suppression and increase T_1 noise in 2D NMR. The most robust tests of phase stability are solids tune-up sequences used for verifying performance for line-narrowing sequences, such as WAHUHA or MREV-8, because these sequences are fairly independent of amplitude stability.

Another test is the 13° test in which two 90° pulses separated by 1 ms are applied with the transmitter placed 37 Hz off-resonance. The resulting NMR response stability is a product of both rf amplitude stability and phase stability because variations in phase between the pulses induce an amplitude change. The observed amplitude error should be divided by a factor of 7.1 to obtain a measure of phase error, in degrees.

Pulse Turn-on Time

- *Experiment* – Single-scan experiments are taken in which the pulse is varied from 0 to 1–2 μs in minimum pulse-width steps at low enough power so that the response is linear. The response is fitted to a straight line and the turn-on time is determined.

  Because of differences in implementation, turn-on times for channel 2 are usually longer than for channel 1, even though the hardware is identical.
- *Purpose* – The quality of modern rf is good enough that examination of pulse shapes using an oscilloscope is not as informative as well-designed and executed NMR tests.

The turn-on and turn-off characteristics of a very short pulse are properties that can be measured sensitively by NMR.

- The turn-on test measures the amplitude of a signal following a short variable-length pulse. In the limit of a small flip angle, this dependence is linear. The data are analyzed and least-squares fitted to a straight line. The intercept is the pulse turn-on time.

Attenuator Linearity

Experiment – For a small flip angle pulse, the rf coarse power is varied from maximum to minimum in single-scan mode. The data are plotted in a horizontal stack to facilitate visual inspection. The data are fitted to a linear regression and plotted in phased mode to show any phase change as a function of power.

Purpose – Overall power control is accomplished using PIN diode-controlled rf attenuators. These attenuators are precision devices that should have negligible phase change throughout their full range. The amplitude response should also be logarithmic. A log regression analysis should show the extent of fit to the ideal. The phase change as a function of power is examined. Raw, uncorrected output should be examined without software adjustment of phase and amplitude.

This test does not permit a full assessment of the cause of the phase error, because the amplifier might be in compression at the maximum power output.

Attenuator Linearity at Reduced Power

Experiment – The attenuator linearity test is repeated but with output of the transmitter reduced by the linear modulator. This is done to isolate the effect of the rf amplifier.

Purpose – The attenuator linearity test is performed, but with reduced power input to the attenuator (using the linear modulator to reduce the output power from the transmitter). Raw, uncorrected output should be examined without software adjustment of phase and amplitude corrections.

Linear Modulator Linearity Tests

Experiment – With the coarse rf amplitude set at a value 23 dB down from maximum, the rf power is varied using the linear modulator. The linear modulator is used for fine power control and shaped rf excitation. The rf amplitude is varied, over a range of 60 dB, in 100 equally-spaced steps over the whole range. This should produce a linear ramp of signal response following a small flip-angle pulse. Spectra are plotted in a horizontal stack of spectra in phased mode with the highest signal full scale. The width of the plotted region around the water is set narrow enough to clearly show the base of the water peak. The data are fitted to a straight line using a linear regression analysis and plotted.

Purpose – Further power control is possible using the linear modulator present on the NMR transmitter board. This test produces a series of experiments in which the pulse power is changed over the full range of the modulator. The linear nature is tested by a linear least-squares fit of the data.

Predictable power control is essential for delivering accurate shaped pulses and for precise power level control in Hartmann-Hahn experiments in both solids and liquids. Raw, uncorrected output should be examined without software adjustment of phase or amplitude.

Linear Modulator Linearity Tests with Attenuators Set to Full Attenuation

Experiment – The linear modulator linearity test is repeated, with the coarse rf amplitude control set for minimum power (resulting in a maximum attenuation of 139 dB). The pulse width is increased correspondingly to obtain comparable signal-to-noise as in the linear

modulator linearity test. The data are fitted to a straight line using a linear regression analysis and plotted.

Pulse Shape Test—DANTE

Experiment – The rf amplitude is set for a 20 µs 90° pulse, and the result is compared to that for a single-scan spectrum:

- 10 pulses, 2 µs each
- 20 pulses, 1 µs each
- 25 pulses, 800 µs each
- 50 pulses, 400 µs each
- 100 pulses, 200 µs each

For all except the first 20 µs pulse, a 1 µs delay is inserted between each pulse. The data are plotted in a horizontal stack to permit comparison of amplitudes. The amplitudes are measured and printed.

Purpose – A DANTE-type test is performed in which the signal response following a 20-µs pulse is measured. This is compared with a series of experiments in which the pulse is increasingly divided into series of pulses spaced by 1 µs. The sum of the pulses is held constant at 20 µs.

If the pulse shape is *ideal* and the total time of the pulse train is short compared to T_2, the rotation of magnetization should be identical. As the pulse length shortens, any non-ideality of pulse shape is revealed as a reduction in intensity.

Phase Switch Settling Time

Experiment – Parameters `p1` and `pw` are set to the same value (1 µs) and 30 spectra are acquired using a 2-pulse sequence, with the first and second pulses separated by a delay of 20 µs. The phase of the second pulse is shifted 180° from the first pulse at a variable time prior to the second pulse. A single-pulse spectrum is also acquired. The arrayed 2-pulse spectra and the single-pulse spectrum are plotted, with the single-pulse spectrum last. This last spectrum serves as a reference. The phase shift should be accomplished in 100 ns or less.

Purpose – This test exercises the phase-shift hardware by finding the time needed to perform a 180° phase shift. The pulse sequence is a version of *jump-and-return* in which two 1-µs pulses are executed just 20 µs apart. Ideally, because the second pulse has a 180° phase shift with respect to the first pulse, there should be no excitation. By varying the time before the second pulse, at which the phase shift is done, from 0 to 20 µs, an estimate of the phase switch and settling time can be made. The last spectrum is that from just a single, 1-µs pulse, and serves as a reference.This phase shift should be accomplished in 100 ns or less.

RF Homogeneity

^{1}H RF Homogeneity Experiment – One hundred experiments are run in which the pulse width is incremented from 1 to 100 µs. The spectra are plotted in a horizontal stack in phased mode and sufficiently expanded so that the base of the water can be examined using the same phase settings for each spectrum (use channel 1).

^{13}C RF Homogeneity Experiment – In the pulse sequence
`delay–pw90(1H)–delay(1/2JCH)–pw(13C)`

the `pw(13C)` is varied from 0 to a flip angle greater than 900° while observing the ^{13}C-coupled protons. The 0-flip-angle spectrum is adjusted to full scale and the data expanded

to show only the ^{13}C-bound protons side-by-side to permit measurement of X-coil rf homogeneity. The results are plotted and displayed in magnitude mode showing one of the lines of the methanol doublet.

Purpose – This test checks the homogeneity of the rf field strength throughout the active region of the sample. In an ideal case, for nuclei having reasonable T_2 values, the signal generated following a 360° + 0° pulse should match that following a 0° pulse. The signal strength as a function of flip angle should be sinusoidal. The amount of drop off is related to the inhomogeneity of the rf field.

High rf homogeneity is important because many important pulse sequences use a large number of pulses. The signal losses accumulate with each pulse such that, in worst cases, all the desired signal is lost. Most heteronuclear, indirect detection experiments on large molecules use HSQC pulse sequence components. These contain 6 to 10 ^{1}H pulses, including 4 to 8 X nucleus 180° pulses. High rf homogeneity is especially important in these cases.

Receiver Test

Experiment – Single scan spectra are collected that span the range of receiver gain and divide that range into at least 25 evenly spaced values of gain, including the highest and lowest gain values.The data are plotted with the highest signal on scale so that the heights can be easily compared.

The results are fitted to a straight line using linear regression, and the fitted data are plotted. Next, the data are normalized and plotted with the water signal held to a constant height so that the noise levels are easily compared (a few mm of noise in the baseline are provided).

The signal-to-noise ratios for the water line in all spectra are measured with a spectral width of 10000 Hz and no oversampling. Channel 1 is used for the acquisition. With oversampling ×10, the experiment is repeated. Processing, plotting, and quantization of the oversampled data are the same as for the data from the 10000-Hz experiment.

Purpose – Receiver gain is selectable in a logarithmic manner (in dB). In an ideal case, variation of receiver gain should produce a logarithmic dependence of signal strength. As the gain is lowered, the noise becomes dominated by noise generated in the ADCs, not in the preamplifier and probe. Regardless of the signal strength, operation in this range of gain will produce poorer signal-to-noise.

Image Rejection Test

Experiment – Plot the data from the following tests first in a horizontal stack, with the single-scan data on-scale, and then with the vertical scale increased 100 times. Quantitate the average image and center glitch.

- Four single-scan and four 4-scan 90° pulse spectra are acquired in which the carrier frequency is shifted 1000 Hz from the water. The carrier position is not changed during the pulse sequence and acquisition, and digital filtering is not used.
- The test is repeated using frequency-shifted quadrature detection (FSQD) with single scans. FSQD is described FSQD is described with the digital filtering information in the *Getting Started* manual.

Purpose – This test checks the inherent balance in the two quadrature channels and the ability of phase cycling to eliminate any quadrature image. Four single-transient and 4 four-transient responses are collected and compared.

Quadrature images can also be eliminated using digital filtering techniques. The FSQD test measures image rejection under these conditions.

Shaped Pulse Test (Channels 1 and 2)

Gaussian-Shaped Pulse Excitation

Experiment – A gaussian-shaped pulse, with excitation bandwidth at 50% amplitude about 200 Hz, is applied (e.g., a 12-ms, 90° pulse length). Single-scan spectra are taken with the transmitter stepped over the range ±250 Hz from resonance, in 5–Hz steps.

The data are plotted in a horizontal stack, with the on-resonance spectrum at full scale to illustrate the gaussian shape of excitation. The vertical scale is increased by ×10 and plotted again to show the wings.

Purpose – The most demanding test of shaped pulse accuracy is the ideality of the NMR data following a shaped pulse. This test determines the accuracy of a gaussian pulse by examination of the off-resonance excitation. This is done by repeating the same single-pulse excitation while varying the transmitter position through a wide range.

A stacked array of data should show the magnitude of excitation as a function of offset from resonance. In the ideal case, this excitation envelope would also be gaussian. Any non-gaussian nature of the pulse, *as delivered to the probe*, would be represented by a convolution of excitation envelopes. For example, if the power were not delivered in a linear manner, producing some rectangular nature, the excitation envelope would have some sin*x*/*x* nature, producing characteristic sinc wiggles. The lack of such non-gaussian behavior is a direct measure of the accuracy with which the hardware can deliver an ideal shape to the nuclei.

Gaussian 90° Pulse Stability

Experiment – The rf 90° pulse stability test is repeated using a gaussian pulse at the same peak power.

The data are plotted in a horizontal stack, with the on-resonance spectrum at full scale to illustrate the gaussian shape of excitation.

Purpose – Modern experiments require very high pulse reproducibility to minimize cancellation residuals and T_1 noise in 2D experiments. This tests amplitude reproducibility by comparing a series of spectra obtained with the signal following a single 90° pulse. The statistical analysis produces an rms deviation, in percent of the average peak height.

Gaussian 13° Phase Error

Experiment – The rf 13° test is repeated using a gaussian pulse at the same peak power as in the phase stability test.

The data are plotted in a horizontal stack, with the on-resonance spectrum at full scale.

Purpose – The rf 13° test can be done using shaped pulses. In this case, a gaussian pulse is used at high peak power.

Gaussian SLP 13° Phase Error (Phase-Ramped Gaussian Pulses)

Experiment – This 13° test is repeated using a phase-ramped gaussian pulses. The rf carrier should be 37 Hz off-resonance from water, but the center of excitation of the gaussian phase-ramped pulses should be 1000 Hz from the carrier. The amplitude of the gaussian pulses is set high enough to exert a 90° pulse on the water.

Purpose – The 13° test can be done using phase-modulated pulses. These types of pulses provide single- or multiple-frequency selective excitation through the use of both amplitude and phase modulation.

Shaped Pulse Settability

Experiment – Single-pulse, single-scan spectra are collected. The rf power is dropped in eight successive spectra by 3 dB each time and the pulse width increased so that a 90° flip angle is maintained. The spectra are plotted in a horizontal stack for easy amplitude comparison.

Purpose – An rf attenuator should permit accurate power control. In this case, the pulse length is repeatedly incremented while appropriately reducing power levels. The NMR response should be identical.

Shaped Pulse Test – Rectangular, Gaussian, and EBURP-1

Experiment – Single-scan, one-pulse excitation spectra are collected using rectangular, gaussian, and EBURP-1 pulses at the same peak amplitude (note the power value and pulse lengths). Constant peak amplitude is maintained; therefore, pulse width ratios of 1.0:2.4:16.0 for the rectangular:gaussian:EBURP-1 pulses, respectively, are used to obtain the same flip angle. Spectra are plotted side-by-side in absolute intensity mode at full vertical scale.

At any constant power, the 90° pulse lengths should reflect their theoretical ratios. Here, the pulse lengths are set in a ratio of 1:2.4:16. The resulting NMR responses should be identical in amplitude.

Shaped Pulse Test—Constant Bandwidth for a Variety of Shapes

Experiment – Single-scan, one-pulse excitation spectra are collected using a variety of shapes that are automatically calculated using Pbox, based on a single pulse calibration using a rectangular pulse, for a constant 4000 Hz bandwidth. The shaped pulses have different peak amplitudes and pulse widths (note the power value and pulse lengths). Spectra are plotted side-by-side in absolute intensity mode at full vertical scale. The resulting NMR responses should be identical in amplitude.

Shaped Pulse Scalability

Experiment – A small flip-angle gaussian pulse is used for a single-transient, one-pulse spectrum. The linear modulator is used to scale down the amplitude of the pulse in 100 steps over a range of 60 dB. Plot widths are set small enough to show the base of the water and plot all spectra in a horizontal stack in phased mode with the maximum signal spectrum at full scale.

Purpose – The linear nature of the system is graphically tested by measuring NMR response when the amplitude is under full control, both by the rf attenuator and by the linear modulator.

^{13}C Test

X-coil rf homogeneity can be determined using an indirect detection pulse sequence. Sensitivity in many indirect detection experiments is markedly affected by X-coil performance because of the large number of 180° pulses used.

X-decoupling is tested for various modulation schemes at constant amplitude (WALTZ-16, GARP-1, etc.) as well as more powerful adiabatic pulse techniques. Efficiency is measured by varying the ^{13}C decoupling frequency while observing the proton spectrum under broadband decoupling.

^{13}C 90° Pulse Width Calibration

The power level for a 90° flip of approximately 15 µs on the X-coil of the probe is determined. Amplifier compression is determined by lowering power by 12 dB and redetermining the 90° pulse width. Both results are reported.

X-Phase Modulation Decoupling Profiles

^{13}C power level is reduced 20 dB from the level used to obtain a 15 µs 90° (approximately 1.8 kHz), and the ^{13}C decoupling efficiency is determined for the following phase-modulated, constant-amplitude broadband decoupling sequences:

- WALTZ-16
- GARP-1
- XY-32
- MLEV-16

The ^{13}C decoupling frequency is varied over a range of ± 80 ppm in a series of single-scan, proton-observe experiments. Only the ^{13}C-bound protons are shown in the expanded spectrum, which is plotted with spectra side-by-side in absolute intensity mode to illustrate decoupling efficiency.

X-Adiabatic Decoupling Profiles

The decoupling profile experiment is repeated with the following adiabatic decoupling schemes: STUD modulation and WURST modulation

Decoupler Heating

The same test as in the variable temperature test is performed but this time using a 75-ms ^{13}C decoupling period prior to acquisition within a total recycle time of 1.5 seconds, including acquisition. One-hundred, single-scan spectra are collected with ^{13}C decoupling followed by 100 identical spectra with no decoupling. The spectra are plotted in a stacked manner to permit examination of the rate of change of temperature, the homogeneity of temperature, and the length of time necessary to reach equilibrium. The rf field strength should be sufficient to decouple over a 160 ppm range using `garp-1`. (Note that decoupling over 160 ppm at ^{1}H frequencies over 750 MHz (200-MHz ^{13}C) require efficient broadband adiabatic decoupling schemes.)

Gradient Tests

Gradient Profile

Experiment – A spectrum with a 100 kHz spectral width is acquired using a gradient echo (collect echo during a Z-axis gradient). This acquisition is repeated for both positive and negative gradients that are sufficient to spread the pattern greater than 50 kHz at the base. Gradient strength and duration as well as the size of the active length of the coil are noted. The experiment is repeated for both the X-axis and Y-axis gradients if available.

Purpose – This test uses pulsed field gradients (PFGs) to quantitate the gradient field strength. The width of the pattern is directly proportional to the gradient strength. The width at 20% of maximum is used to calculate the gradient strength. Both positive and negative gradients are used. This should be done for all orthogonal axes available.

Field Recovery Stability

Experiment – The 90° pulse stability test is performed but, preceding the rf pulse, is a 1-ms, 30-G/cm Z-axis gradient pulse, which is then followed by a 100–µs field stabilization

delay. This test is repeated with a 10 G/cm gradient pulse. If X and Y gradients are available, the test is repeated with a 10 G/cm gradient pulse for each gradient.

Purpose – A gradient is applied prior to a measuring pulse. The stability of the signal response is used to measure the ability and reproducibility of the system to recover from the gradient pulse.

Field Recovery

Experiment – The gradient 90° pulse stability test is repeated with the field recovery delay varied from 0 to 1000 µs, using a positive 10 G/cm Z-axis gradient pulse. The spectra are plotted in a horizontal stack with the 1000 µs data at full scale. The test is repeated using a negative 10 G/cm gradient and, if available, for both the X and Y gradients. Rectangular and half sine gradients are used. Recovery is defined as the time it takes to recover to 95% or more of the amplitude.

Purpose – A gradient is applied prior to a measuring pulse and the time before the pulse is varied. The rate of recovery determines how soon a pulse may be applied.

Gradient Echo Stability

Experiment – The 90° pulse stability test is run, this time with a positive gradient for 1 ms, a 500 µs delay, and a negative gradient for 1 ms following the rf pulse. The following gradient strengths and axes are used.

- 30 G/cm—Z axis only
- 10 G/cm—Z axis and, if present, X and Y axes

Purpose – Following a single pulse, a pair of opposite-signed gradients is applied. The stability of the resulting refocused signal measures any instability in the gradient amplitude, as well as the accuracy of the gradient level control. This should be done for all orthogonal axes available.

Gradient Effect on Cancellation Test

Experiment – Four 1-scan, four 2-scan and four 4-scan 90° pulse spectra are acquired, with a 10 G/cm Z-axis gradient pulse 100 µs prior to the rf pulse and the transmitter phase held constant while the receiver is phase-cycled 0, 2, 1, 3. The spectra are plotted in a horizontal stack with the single-scan spectra on scale. Vertical scale is increased by 100 times and plotted in the same manner. The average residual signal for the 4-scan cancellation is quantitated and the results plotted and analyzed as above.

Purpose – The cancellation test is done with a gradient pulse applied 100 µs before the rf pulse. If the lock circuitry and field recovery characteristics are favorable, no deterioration in cancellation efficiency should be noted.

CPMG T_2

Experiment – A CPMG T_2 experiment is performed with 2 ms between 180° pulses for total echo pulse trains ranging from a few milliseconds out to at least $2*T_1$. This experiment is repeated for the case where 500 µs 10 G/cm rectangular pulses are placed around the 180° pulses in each echo, as well as the case in which no gradients are used. The values for T_2 are reported for all cases. The experiment is repeated for the case of a 1% mismatch in gradient amplitude.

Purpose – Following a single pulse, pairs of same-signed gradients are applied within the echoes of a CPMG T_2 pulse echo train. The measured T_2 gives a measure of any instability in the gradient amplitudes as well as the accuracy of the gradient level control. This measurement is compared to an identical experiment in which the gradient amplitudes are

set to zero or mismatched by 1%. More rapid diffusion at higher temperature will cause the gradient/no gradient comparison to worsen. Therefore, performance over time should be compared for the same temperature. (Measurements done at ~4° C have showen no difference in measured T2 for the gradient/no gradient cases, indicating that diffusion is responsible for the difference in these cases at higher temperatures).

Other Tests

Heating During Spin Lock Test

Experiment – The same test as in the variable temperature test is performed using a 70-ms ^{1}H pulse at an rf field strength of 10 kHz with a total recycle time of 1.5 seconds, including acquisition. The data are plotted in the same way as in the VT test.

Purpose – Many modern experiments use spin locks or decoupling within their pulse sequences. Often, rf fields can cause significant sample heating. Depending on the nature of the probe, this heating can be a problem causing baseline artifacts and t_1 noise. It is important to quantify the amount of sample heating, the speed in attaining a new equilibrium temperature, the homogeneity of temperature throughout the sample during the heating period, and the final change in temperature.

This test imposes a rather strong (10 kHz) rf field for a period of time often used in TOCSY experiments, using a recycle time of 1.5 seconds (including acquisition). Single transients are acquired, at a rate of one per 1.5 seconds. The data show any temperature change at the expected 0.01 ppm/degree. The intensity and/or linewidth can be used to measure temperature homogeneity. The number of transients needed to attain a new equilibrium temperature measures the ability of the probe to stabilize the effects of internal sample heating. The final shift value indicates the total temperature change. This can be used to reduce the requested temperature value so as to obtain the desired equilibrium. Of course, the amount of heating is a function of the sample itself, primarily its salt content. The same approach may be used to follow the actual temperature in the sample under the influence of X-nucleus decoupling.

Lock Tests

Experiment – Lock power is varied over a 30 dB range and the lock level recorded. The experiment is repeated for the lock gain. A log regression analysis is performed to confirm the relationship between the lock signal and power/gain.

Purpose of Lock Gain Test – Lock gain is selectable in a logarithmic manner (in dB). In an ideal case, variation of receiver gain should produce a logarithmic dependence of signal strength.

Purpose of Lock Power Control Using an RF Attenuator – Overall lock power control is accomplished using computer-controlled rf attenuators. The amplitude response should also be logarithmic. A log regression analysis should show the extent of fit to the ideal.

Spectral Purity Test

Experiment – Four single-scan 100 kHz spectral width spectra are acquired with no pulse. Each spectrum is plotted with a few millimeters of noise.

Purpose – RF purity of the transmitter and receiver can be tested by recording data without any excitation pulse. The spectrum reveals any artifactual signals.

Variable Temperature Test

Experiment – Single-scan spectra are acquired during an increase of 5°C in sample temperature. Spectra are recorded sequentially. Spectra are taken every 2 seconds until the

sample reaches equilibrium, as reflected in a stable chemical shift of a methyl proton. Spectra are plotted in a stacked manner to permit examination of the rate of change of temperature, the homogeneity of temperature, and the length of time necessary to reach equilibrium.

Purpose – The sample temperature is increased by 5^{o} C while recording spectra every 2 seconds. Most methyl resonances show a chemical shift of `(sfrq/100)` Hz/^{o}C and this shift, therefore, indicates the actual temperature distribution within the sample. The methyl resonance should move quickly and homogeneously to its new equilibrium position. The rate of change and homogeneity of change demonstrate the VT performance of the probe and regulation hardware.

Small-Angle Phase Shift Test

Experiment – Single-scan spectra are acquired in which the phase of the pulse is incremented by 10° in each spectrum through a full 360° at constant receiver phase. Spectra are plotted in a horizontal stack to show a smooth phase rotation of the spectrum. The test is repeated using pulses generated from channel 2.

Purpose – Small-angle phase adjustment is used in multiple-quantum selection (`q>2`), phase-modulated pulses, and a variety of complex pulse sequences. This test exercises the phase-shift hardware by varying the pulse phase in small increments over 360°.

1.8 Varian Sales Offices

For product sales and service information, contact one of the Varian sales offices:

- Argentina, Buenos Aires, (114) 783-5306
- Australia, Mulgrave, Victoria, (3) 9566-1138
- Austria, Vösendorf, (1) 699 96 69
- Belgium, Brussels, (2) 721 48 50
- Brazil, Sao Paulo, (11) 829-5444
- Canada, Ottawa, Ontario, (613) 260-0331
- China, Beijing, (10) 6846-3640
- Denmark, Herlev, (42) 84 6166
- France, Orsay, (1) 69 86 38 38
- Germany, Darmstadt, (6151) 70 30
- Italy, Milan, (2) 921351
- Japan, Tokyo, (3) 5232 1211
- Korea, Seoul, (2) 3452-2452
- Mexico, Mexico City, (5) 523-9465
- Netherlands, Houten, (30) 635 0909
- Norway, Oslo, (9) 86 74 70
- Russian Federation, Moscow, (95) 241-7014
- Spain, Madrid, (91) 472-7612
- Sweden, Solna, (8) 445 1601
- Switzerland, Zug, (41) 749 88 44
- Taiwan, Taipei, (2) 2698-9555

- United Kingdom, Walton-on-Thames, England (1932) 898 000
- United States, Palo Alto, California,
  Varian, Inc., NMR Systems
  Customer Sales Support, (650) 424-5145
  Service Support, Palo Alto, California, **1 (800) 356-4437**
  E- mail: customer.support@nmr.varian.com

  North American Service Manager
  6440 Dobbin Rd, Ste D, Columbia, MD 21045
  (410) 964-3065
- Venezuela, Valencia (41) 257608

1.9 Posting Requirements for Magnetic Field Warning Signs

The strong magnetic fields that surround a superconducting magnet are capable of causing death or serious injury to individuals with implanted or attached medical devices such as pacemakers or prosthetic parts. Such fields can also suddenly pull nearby magnetic tools, equipment, and dewars into the magnet body with considerable force, which could cause personal injury or serious damage. Moreover, strong magnetic fields can erase magnetic media such as tapes and floppy disks, disable the information stored on the magnetic strip of automated teller machine (ATM) and credit cards, and damage some watches.

To warn of the presence and hazard of strong magnetic fields, the customer is responsible for posting clearly visible signs warning of magnetic field hazards. This responsibility includes measuring stray fields with a gaussmeter.

Radio-frequency emissions may also pose a danger to some individuals. The rf emission levels from Varian NMR equipment have been measured and compared to the IEEE/ANSI C95.1-1991 standard. For further information, refer to the *RF Environment* section of the *Installation Planning Guide*.

Warning Signs

Varian provides signs to help customers meet this posting responsibility. These signs *must* be posted according to the following requirements *before the magnet is energized*:

1. *10-gauss warning signs* (Figure 1) – Post along the 10-gauss perimeter of the magnet so that a sign can be easily seen by any person about to enter the 10-gauss field from any direction. Refer to the manuals supplied with the magnet for the size of a typical 10-gauss stray field. Check this gauss level after the magnet is installed.

   Note that the stray field may extend vertically to adjacent floors, and additional signs may be needed there. A sign is not required if the 10-gauss field extends less than 30 cm (12 in.) beyond a permanent wall or less than 61 cm (24 in.) beyond the floor above the magnet.

2. *5-gauss warning signs* (Figure 2) – Post along the 5-gauss perimeter of the magnet so that a sign can be easily seen by any person about to enter the 5-gauss field from any direction. Refer to the manuals supplied with the magnet for the size of a typical 5-gauss stray field. Check this gauss level after the magnet is installed. Note that the stray field may extend vertically to adjacent floors, and additional signs may be needed there.

3. *Magnet area danger signs* (Figure 3) – Post at each entrance to the magnet area. Be sure each sign is outside the 5-gauss perimeter.

10-GAUSS WARNING
STRONG MAGNETIC FIELD
Tools and Equipment
Strong magnetic fields are present that can make magnetic items suddenly fly towards the magnet, which could cause personal injury or serious damage. Do not take tools, equipment, or personal items containing steel, iron, or other magnetic materials closer to the magnet than this sign.
Dewars
The stray field of the magnet can pull a magnetic dewar into the magnet body, causing serious damage. Use only nonmagnetic stainless steel dewars. Do not use iron or steel dewars during servicing.
VARIAN

Figure 1. 10-Gauss Warning Sign

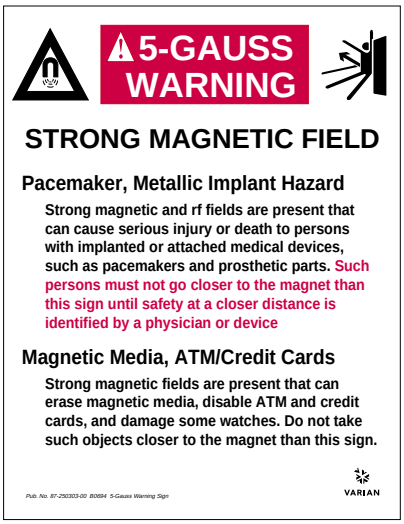
5-GAUSS WARNING
STRONG MAGNETIC FIELD
Pacemaker, Metallic Implant Hazard
Strong magnetic and rf fields are present that can cause serious injury or death to persons with implanted or attached medical devices, such as pacemakers and prosthetic parts. Such persons must not go closer to the magnet than this sign until safety at a closer distance is identified by a physician or device
Magnetic Media, ATM/Credit Cards
Strong magnetic fields are present that can erase magnetic media, disable ATM and credit cards, and damage some watches. Do not take such objects closer to the magnet than this sign.
VARIAN

Figure 2. 5-Gauss Warning Sign

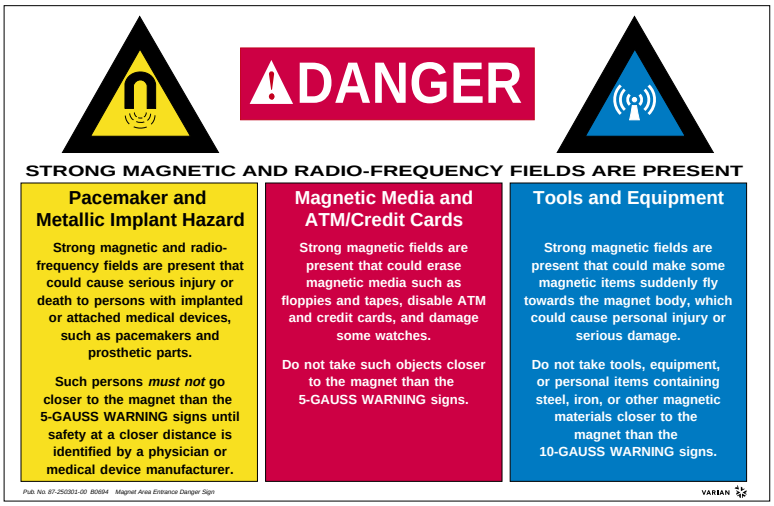
DANGER
STRONG MAGNETIC AND RADIO-FREQUENCY FIELDS ARE PRESENT
Pacemaker and Metallic Implant Hazard
Strong magnetic and radio-frequency fields are present that could cause serious injury or death to persons with implanted or attached medical devices, such as pacemakers and prosthetic parts.
Such persons must not go closer to the magnet than the 5-GAUSS WARNING signs until safety at a closer distance is identified by a physician or medical device manufacturer.
Magnetic Media and ATM/Credit Cards
Strong magnetic fields are present that could erase magnetic media such as floppies and tapes, disable ATM and credit cards, and damage some watches.
Do not take such objects closer to the magnet than the 5-GAUSS WARNING signs.
Tools and Equipment
Strong magnetic fields are present that could make some magnetic items suddenly fly towards the magnet body, which could cause personal injury or serious damage.
Do not take tools, equipment, or personal items containing steel, iron, or other magnetic materials closer to the magnet than the 10-GAUSS WARNING signs.
VARIAN

Figure 3. Magnet Area Danger Sign

Stray magnetic fields can reach beyond the published distances when two or more magnetic fields intersect or when the field extends over large ferromagnetic masses or structures (steel doors, steel construction beams, etc.). In this case, the customer *must* measure the stray field using a gaussmeter to determine how the 5- and 10-gauss fields are altered (contact a scientific instrumentation supplier for information on acquiring a gaussmeter).

You can request additional signs from Varian by telephoning 1-800-356-4437 in the United States or by contacting your local Varian office in other countries.

Chapter 2. Console and Magnet Test Procedures

Sections in this chapter:

- 2.1 “Automated Test Procedures—Running AutoTest” this page
- 2.2 “Manual Test Procedures Required to Demonstrate Console Operation” page 42
- 2.3 “Procedures for Contracted Custom Console Specifications” page 48

This chapter contains the procedures for testing UNITY*INOVA* NMR consoles and magnets. These procedures are required to demonstrate the specifications listed in Chapter 3, “Consoles and Magnets Specifications,” for UNITY*INOVA* NMR consoles and magnets.

2.1 Automated Test Procedures—Running AutoTest

The AutoTest automated test procedures software features a series of automated tests for both the console and the probe. These tests demonstrate the console performance and its compliance with the acceptance test specifications as well as some of the probe acceptance tests and its compliance with the specifications listed in Chapter 3. AutoTest tests only channels 1 and 2.

The automated tests write the results to a text file and plot the resulting spectra. Although plotting is optional, plotting should be activated for acceptance testing and customer review. A hard copy of the AutoTest report should be attached to the appropriate acceptance test results in Chapter 4.

Refer to “Details of AutoTest Experiments,” page 24, for detailed descriptions of the tests performed by AutoTest.

Installing Autotest

AutoTest is available beginning with the VNMR 6.1B. AutoTest can be installed at any time, but it is most convenient to install it when VNMR is installed. If AutoTest is not already installed, use these steps to install it now.

CAUTION: **Remove any files used for previous versions of AutoTest from your `vnmrsys` directories, particularly any `~/vnmrsys/seqlib/AT*` or `~/vnmrsys/maclib/AT*` files. Remove `AT*.DEC`, `gauss32.RF`, `gauss.RF` and `eburp1.RF` from `~/vnmrsys/shapelib`, if present. Make sure that `~/vnmrsys/maclib/autotest` is not present.**

1. Mount the VNMR CD-ROM, change to the CD-ROM directory, and enter `./load.nmr`. The VNMR installation menu appears.
2. Select the UNITY*INOVA* system at the top of the menu. The VNMR installation menu for UNITY*INOVA* appears.

3. Select the AutoTest checkbox. Click on **install**.

Refer the manual *VNMR and Solaris Software Installation* for further details.

Sample for AutoTest

As the sample, AutoTest uses 0.1% ^{13}C-enriched methanol in 1% H_2O/99% D_2O. The sample is doped with gadolinium chloride at a concentration of 0.30 mg/ml, which produces a ^{1}H T_1 relaxation time of about 50 to 75 ms. The resulting line width is considerable larger than the magnet-determined line width because of the paramagnetic relaxation contribution.

Setting Up for AutoTest

To set up for autotest:

1. Make sure all units of the rf system (transmitters, linear modulators, rf attenuators, amplifiers, receivers, and probes) are in the standard configuration.
2. If the system has a PFG accessory installed, make sure that the gradient amplifier is on and **`pfgon`** is set correctly for the number of gradients available:
   - `pfgon='nny'` for Z-axis only.
   - `pfgon='yyy'` for triax.

   Allow sufficient time for stabilization. AutoTest calibrates the gradients.
3. Insert the AutoTest sample.
4. Set the VT to 25° C. Allow the temperature of the sample to regulate and equilibrate. While specifications are determined at 25° C, normal day-to-day AutoTest runs may be done at other temperatures.
5. Tune the probe, and lock on the D_2O resonance.
6. Shim the field on the sample to give a *nonspinning* half-height that is dominated by the paramagnetic relaxation agent. Because the H_2O line is quite broad, this should not require much time.
7. Note the **`tpwr`** value necessary to produce a ^{1}H 90° pulse width that is within specification (8 to 10 µs), but does not cause probe arcing.

   AutoTest uses this `tpwr` value in determining the ^{1}H 90° pulse width and calculates the amplifier compression at this `tpwr` value.
8. To save the results of a previous AutoTest run, rename the `history` and `data` directories. For further information, refer to the manual *System Administration*.

Running AutoTest

To run AutoTest, you enter values and make selection in the AutoTest window and then select the Begin Test button. Be sure to press the Return key after entering each value.

1. Enter the macro **`autotest`**. The AutoTest window opens, similar to Figure 4. .
2. Select the **Configuration** tab at the top of the window, if not already selected.
3. Fill in the fields for the `Operator Name`, `Console type`, etc. at the top of the window. In the `pw90` field, enter an approximate ^{1}H `pw90` for the value of `tpwr` that you entered (10 µs is recommended)

Figure 4. AutoTest Program

4. Select the options desired in the checkboxes in the middle and lower part of the window.

   A full AutoTest run including all available options must be run before any single test or partial set of tests is specified. Most of these tests rely on calibrations that are performed as part of the full AutoTest run. This option is specified by the *All Tests* checkbox.

5. Click the **Begin Test** button at bottom of the window to start the test(s).

   AutoTest begins. The total time for the test(s) depends on the test(s) specified and on plotting, and CPU speed.

   As AutoTest runs, FIDs are stored in the `data` directory and the results from the tests are stored in the `history` directory.

6. After AutoTest finishes, inspect the reports and compare the results to the specifications.

Saving Data and FID Files from Previous Runs

As AutoTest executes, data and FID files are written into the `history` and `data` directories, which are located in the `autotest` directory. The `autotest` directory is usually located in the directory `vnmrsys` of a user's home directory. The contents of the `data` directory are progressively overwritten as AutoTest continues.

Before starting a new AutoTest run, do the following to save the data from a previous run:

1. Open a UNIX window. and enter **`cd ~/vnmrsys/autotest`**.
2. Change the name of the `history` directory by entering, for example, **`mv history history.old`**.

3. Change the name of the `data` directory by entering, for example,
   `mv data data.old`

Creating Probe-Specific Files

If you run AutoTest with different probes, you might want to keep separate `autotest` directories. Use the following steps to create probe-specific files.

1. After you have run AutoTest using a specific probe, change the name of the `autotest` directory by using the `mv` command, for example:
   `cd ~/vnmrsys`
   `mv autotest autotest_`*`probe_1`*
   Where *`probe_1`* is the name of the probe that was tested, for example, `5mmTriplePFG` or `5mmID`.

   Any new AutoTest run automatically creates a new `autotest` directory in the user's `vnmrsys` directory. The only file that needs to be updated would be **`~/vnmrsys/autotest/parameters/standard.par`**. This can either be copied from the saved `autotest` file or the parameter set may be retrieved using `rt` or `rtp`, the parameters updated and then saved, replacing the `standard.par` file. This should be safe for any parameters displayed in the `dg` window, but there are several parameters dealing with gradients and indirect detection that must also be checked. It is safest to do an All Tests run the first time a new probe is used.

   Once a calibrated `standard.par` parameter set is present, `autotest` directories may be renamed whenever a probe is changed. In this way, `history` files may be kept specific to a probe.

2. To change the file name back to `probe_1` (or the name you have chosen), enter, for example:
   `cd ~vnmrsys`
   `mv autotest autotest_`*`probe_2`*
   Where *`probe_2`* is the name you have chosen for the probe last tested.
   `mv autotest_`*`probe_1`* **`autotest`
   Where *`probe_1`* is the name you have chosen for the probe you now want to test.

   If you need to repeat any individual test, you can do so by recalling the appropriate FID from the `data` directory. The experiment can then be started with the `go` command without overwriting the previous data. Or the test may be selected from the Test Library after using the `autotest` macro or a menu calling this macro.

Tests Performed by AutoTest

This section provides an outline of the tests run by AutoTest. A full description of each test is provided in "AutoTest Automated Instrument Testing," page 15.

Automated Console Acceptance Tests

AutoTest performs the following automated console acceptance tests:

- 90° pulse stability channel 1 and channel 2
- 30° amplitude stability channel 1 and channel 2
- Pulse turnon time channel 1 and channel 2
- Phase cycle cancellation (2-scan test is run as demonstration, no specification is set)
- Quadrature image: 1 scan and 4 scans

- Frequency-shifted quadrature image: 1 scan
- Phase stability test (13° test) channel 1 and channel 2
- Attenuator test channel 1 and channel 2

  Full power correlation coefficient and standard deviation (standard deviation is provided for reference, no specification is set)

  Reduced power correlation coefficient and standard deviation
- Modulator linearity channel 1 and channel 2

  `tpwr=40`: standard deviation.
  `tpwr=-16`: standard deviation.
- Temperature increase in spinlock test
- Lock power test correlation coefficient
- Lock gain test correlation coefficient
- Variable temperature test

Automated Console Acceptance Tests Using Shaped RF

AutoTest performs the following automated acceptance tests with shaped rf:

- Gaussian 90° stability, channel 1 and channel 2
- Gaussian phase stability test, channel 1 and channel 2
- Gaussian SLP phase stability test, channel 1 and channel 2

Automated Heteronuclear Decoupling Performance Tests

AutoTest performs the following automated decoupling performance tests:

- ^{13}C phase modulation decoupling profiles:
  ^{13}C GARP decoupling profile
  ^{13}C WALTZ-16 decoupling profile
- ^{13}C XY32 decoupling profile
  ^{13}C MLEV-16 decoupling profile
- ^{13}C adiabatic decoupling profiles (if waveform generator is present on the decoupling channel):
  ^{13}C STUD decoupling profile
  ^{13}C WURST decoupling profile
- Sample heating during ^{13}C broadband decoupling

Automated Probe Performance Tests

Note: Individual probe acceptance test procedures and specifications specify probe acceptance tests and specifications. The automated tests do not form the basis of a probe's acceptance or rejection.

AutoTest performs the following 90° pulse width calibrations (PW90):

- ^{1}H 90° pulse width calibrations on channels 1 and 2
- ^{13}C 90° pulse width calibrations (PW90)

AutoTest performs the following rf homogeneity tests:

- ^{1}H rf homogeneity test
- ^{13}C rf homogeneity test

AutoTest performs the following gradient calibrations and performance tests:

- Gradient level for 10 G/cm along the following:
  Z axis for all gradient probes
  X axis for triax probes
  Y axis for triax probes
- Gradient echo stability for the following:
  Z axis at 30 G/cm
  X axis at 10 G/cm
  Y axis at 10 G/cm
  Z axis at 10 G/cm
- Gradient recovery stability for the following:
  X axis at 10 G/cm
  Y axis at 10 G/cm
  Z axis at 10 G/cm
- Gradient recovery for the following:
  X axis at ± 10 G/cm
  Y axis at ± 10 G/cm
  Z axis at ± 10 G/cm
- Cancellation after gradient.

Automated Console Acceptance Demonstration Tests

AutoTest performs the following console demonstration tests:

- High-band amplifier compression
- Low-band amplifier compression
- Temperature rise in decoupler heating test
- AutoGain result for 90° pulse.
- Receiver gain (normal sampling 10-kHz sweep width)
- Receiver gain (oversampling 100-kHz sweep width)
- Folded noise reduction with large spectral width
- Phase switch settling time

2.2 Manual Test Procedures Required to Demonstrate Console Operation

This section contains the required manual test procedures:

- "Homonuclear Decoupling," this page
- "Lock Frequency Stability," page 43
- "Basic Variable Temperature Operation," page 44
- "Magnet Drift," page 45
- "WALTZ 1H Decoupling—Preprogrammed Phase Modulation," page 46
- "WALTZ 1H Decoupling—High-Performance RF Waveform Generator," page 47

For optional or custom-contracted tests, refer to "Procedures for Contracted Custom Console Specifications," page 48.

Homonuclear Decoupling

Sample

0.1% ethylbenzene, 0.01% TMS, 99.89% deuterochloroform ($CDCl_3$)

Part No.	*Sample Tube*
00-968120-70	5 mm
00-968123-70	10 mm

Probe and Hardware Requirements

A 5-mm probe capable of ^1H direct observe is recommended.

Test Procedure:

1. Enter **`rtp('/vnmr/tests/H1sn') su`** to retrieve the test parameter set to the current experiment. Check that the ^1H quarter-wavelength cable is installed (200-MHz, 300-MHz, and 400-MHz systems) and the probe is tuned.
2. Enter **`nt=1 dm='n'`** and **`ga`** to acquire a normal spectrum without decoupling.
3. Move the cursor to the central line of the triplet, and enter **`sd`** to move the decoupler to that frequency.
4. Enter **`dm='n','y' dpwr=20 homo='y'`** and **`ga`** to acquire two spectra; the first spectrum is without decoupling, and the second is with homodecoupling.

   The best values of `dpwr` must be found by experiment. Start with the `dpwr=20`. Too much power can show increased noise; too little fails to decouple the quartet.

Lock Frequency Stability

The stability of the lock is judged by the phase response of a Hahn echo. Run 20 small, single-scan echo experiments at 1 minute intervals and record the phase variations.

Sample

Doped 2-Hz H_2O/D_2O (0.1 mg/ml $GdCl_3$ in 1% H_2O in D_2O), Part No. 01-901855-01 for 5-mm samples.

Probe and Hardware Requirements

A 5-mm probe capable of ^1H direct observe is recommended. This test also requires a Lock Module on the spectrometer.

Test Procedure

1. Check that the ^1H quarter-wavelength cable is installed (200-, 300-, and 400-MHz systems), the probe is tuned, ^1H linewidth shimmed to within 2.5 to 3.5 Hz at the 50% level, and the 90° pulse width determined for the 1% H_2O/99% D_2O sample. Set `pw` to the 90° pulse width value.

2. Enter **`rtp('/vnmr/tests/shmd2o')`** to retrieve the test parameter set to the current experiment. Acquire a normal spectrum and shim the water signal to less than 3-Hz linewidth at 50%.
3. Place the cursor close to the water signal and enter **`movetof`** to move the transmitter offset to within 50 Hz of the water peak.
4. Set the following parameters: **`hom2dj ph sw=2000 lb=1.0 spin='n' d2=1.000 ni=0 sb='n' fn=32k nt=1 ss=0 at=1`**.
5. Set **`pw`** to the 90° pulse width value, **`p1`** to the 180° pulse width value.
6. Create an array of 20 `d1` values by entering **`array('d1',20,60,0)`**.
7. Enter **`ga`** to acquire the spectrum. Phase the first spectrum by entering **`ds(1)`** to display the first spectrum of the array and by entering **`aph0`** to apply a first-order phase correction to the spectrum.
8. Enter **`ai`** to scale all of the spectra to the same vertical scale and enter **`dssh`** to display the arrayed spectra horizontally.
9. Enter **`analyz`** to use the `analyz` macro to process the 20 spectra for average height, phase deviation, and frequency shift.

   The lock frequency stability is displayed as the standard deviation (degrees). When the prompt `Plot Results? (n/y)` appears, answer **`y`** to print the results.

Basic Variable Temperature Operation

For the basic variable temperature (VT) accessory (Varian Part No. 00-992957-00), this test demonstrates that the VT accessory and probe go to the desired temperature as registered on the window of the VT controller. VT accessory operation is described in detail in the VNMR manuals. For the high-stability VT accessory, refer to “Stability Calibration for High-Stability VT Accessory,” page 50.

Dry nitrogen is required as the VT gas if the requested temperature is over 100° C or below 10° C. Otherwise, air can be used. For temperatures below –40° C, dry nitrogen gas is recommended for cooling the bearing, spinner, and decoupler. This gas prevents moisture condensation in the probe and spinner housing.

CAUTION: **The use of air as the VT gas for temperatures above 100° C is not recommended. Such use destructively oxidizes the heater element and the thermocouple.**

Demonstration Limitations

The VT range varies from probe to probe. VT will be demonstrated only at temperatures within the specified range of the probe in use.

If dry nitrogen gas and liquid nitrogen are not available at the time of installation, the range of VT demonstration is limited to temperatures between 30°C and 100°C.

Sample

An empty sample tube with spinner.

Probe and Hardware Requirements

Any variable temperature probe.

Test Procedure

1. Set N_2 gas flow to 9.5 to 10.0 LPM. For temperatures below –100° C, set N_2 flow to 12 LPM.
2. Enter a value for **`temp`**, then enter **`su`**. For values below room temperature, the heat exchanger must be in place. Maintain the requested temperature for 5 minutes.
3. Operate the VT unit within the specifications of the probe. Test the temperature at set points that correspond to the following:
   - Maximum, minimum, and midpoint of the allowed temperature. For example, probes with a VT range of –150 to 200 could be tested as follows: 95, 80, 60 if air is used; 120, 30, 20 if dry nitrogen is used; 120, –100, 40 if a heat exchanger is used.
   - Ambient temperature.

   Adjust the temperature by no more than 50°C, enter `su`, and wait for the temperature to equilibrate.

Magnet Drift

The magnet drift test is an overnight test.

Sample

For 200- and 300-MHz systems, use ^{1}H lineshape sample 20% $CHCl_3$ in 80% deuteroacetone $(CD_3)_2CO$, Part No. 00-968120-76.

For 400-, 500-, 600-, 750-, and 800-MHz systems, use 1% $CHCl_3$ in 99% deuteroacetone $(CD_3)_2CO$, Part No. 00-968120-89

Probe and Hardware Requirements

A 5-mm probe capable of ^{1}H direct observe is recommended.

Test Procedure

1. Enter **`rtp('/vnmr/tests/H1lshp')`** to retrieve the test parameter set to the current experiment.
2. Check that the ^{1}H quarter-wavelength cable is installed (200-MHz, 300-MHz, and 400-MHz systems) and the probe is tuned. Acquire a normal spectrum and shim the chloroform signal to less than 1 Hz linewidth at 50%.
3. Connect to the **`acqi`** window, turn the lock off, turn the spinner off, and set the spinner speed to 0. Make sure the lock signal is on-resonance (the lock signal display should be flat). Disconnect the `acqi` window.
4. Enter **`in='n' spin='n' nt=1 array('d1',11,3600,0) d1[1]=60`**.

   This sets up an array of `d1` values, with the first spectrum to be collected after 1 minute and subsequent spectra to be collected at one-hour intervals.
5. Enter **`ga`** to acquire the spectra.

   The test takes about 10 to 11 hours to finish.
6. After the spectra are acquired, phase the first spectrum by entering **`ds(1)`** to display the first spectrum of the array and by entering **`aph0`** to apply a first-order phase correction to the spectrum.

7. Enter **ai** to scale all of the spectra to the same vertical scale, and enter **dssa** to display the arrayed spectra stacked vertically.
8. Compare the frequency shift of the chloroform peak of the arrayed spectra to the frequency of the first spectrum in the array.

WALTZ ^{1}H Decoupling—Preprogrammed Phase Modulation

Sample

Doped 40% p-dioxane in benzene-d_6.

Part No.	*Sample Tube*
00-968120-91	5-mm
00-968123-91	10-mm

Probe and Hardware Requirements

A 5-mm or 10-mm broadband observe, ^{1}H decouple probe is recommended (i.e. 5-mm or 10-mm broadband probes, 5-mm switchable probes, or 4-nucleus Auto•nmr probes). This test requires the results from the γH_2 calibration that gives a `dpwr` of 1 watt at the probe.

Test Procedure

1. Enter **`rtp('/vnmr/tests/gamah2')`** **`su`** to retrieve the test parameter set to the current experiment. Check that the appropriate quarter-wavelength cable for ^{13}C is installed and the probe is tuned.
2. Set **pw** to the ^{13}C 90° pulse width value and **tpwr** to the power value used to achieve the ^{13}C 90° pulse width.
3. Enter **`dmm='nny' dpwr=30 d1=10`** (for the doped ASTM) or **`d1=30`** (for the dioxane/deuterobenzene sample) and **ga** to acquire the spectra.

   Each spectrum shows a triplet. Using two cursors, measure the distance in Hz between the center peak and one of the outer peak. Use the same outer peak (either the one on the right or the one on the left) for the measurements for both spectra. The two measurements, in Hz, are used as input for the `h2cal` command, which calculates the γH_2 for a given decoupler power level.
4. Enter **`h2cal`** to calculate γH_2. Record the result.
5. Set **dmf** to the $4*\gamma H_2$ value. Set **`dmm='w' lb=1`** and enter the command **`array('dof',9,-2000,500)`** to set up the experiment. Enter **ga** to acquire the data. Enter **`ds(1) aph0 dssh`** to phase and display the spectra. To plot the spectra and acquisition parameters, enter **`pap pl('all') page`**.

WALTZ ^{1}H Decoupling—High-Performance RF Waveform Generator

Sample

Doped 40% p-dioxane in benzene-d_6.

Part No.	*Sample Tube*
00-968120-91	5-mm
00-968123-91	10-mm

Probe and Hardware Requirements

A 5-mm or 10-mm broadband observe, ^{1}H decouple probe is recommended (i.e. 5 mm or 10 mm broadband probes, 5 mm switchable probes, or 4-nucleus Auto•nmr probes). This test requires the presence of waveform generator boards. This test also requires the results from the γH_2 calibration that gives a `dpwr` of 1 watt at the probe.

Test Procedure

If you completed the previous test ("WALTZ 1H Decoupling—Preprogrammed Phase Modulation," page 46), skip to step 5 in the following procedure.

1. Enter **`rtp('/vnmr/tests/gamah2') su`** to retrieve the test parameter set to the current experiment.
2. Check that the appropriate quarter-wavelength cable for ^{13}C is installed and the probe is tuned.
3. Set pw to the ^{13}C 90° pulse width and **`tpwr`** to the power used to achieve the ^{13}C 90° pulse width
4. Set **`dm='nny' dpwr=30 d1=10`** (for the doped ASTM) or **`d1=30`** (for the dioxane/deuterobenzene sample), and enter **`ga`** to acquire the spectra.

   Each spectrum shows a triplet.
5. Using two cursors, measure the distance in Hz between the center peak and one of the outer peak. Use the same outer peak (either the one on the right or the one on the left) for the measurements for both spectra. The two measurements in Hz are used as input for the `h2cal` command, which calculates the γH_2 for a given decoupler power level.
6. Enter **`h2cal`** to calculate γH_2. Record the result.
7. Set **`dmf`** to 4* γH_2 value. Set **`dmm='ccp' dseq='waltz16' dres=90 lb=1`** and enter **`array('dof',9,-2000,500)`** to set up the experiment using the first decoupler.

   If it is the second decoupler that is capable of programmable decoupling using the waveform generator, set **`dmm2='ccp' dseq2='waltz16' dres2=90`**.

   Enter **`ga`** to acquire the data. Enter **`ds(1) aph0 dssh`** to phase and display the spectra. To plot the spectra and acquisition parameters, enter **`pap pl('all') page`**.

2.3 Procedures for Contracted Custom Console Specifications

The following procedures are described in this section:

- "Temperature Accuracy for VT Systems," this page
- "Stability Calibration for High-Stability VT Accessory," page 50
- "Homospoil Demonstration," page 51
- "Sucrose Anomeric 1H Signal-to-Noise Ratio," page 52
- "Aqueous Phenylalanine Water Suppression," page 54

These procedures are not required as part of the acceptance testing but may be performed during the installation of the Varian NMR spectrometer system if mutually agreed to before the installation. Users are encouraged to perform these procedures on their own as further familiarization and evaluation of the instrument performance.

Temperature Accuracy for VT Systems

The tests in this section check temperature accuracy calibrations for high and low temperatures using ethylene glycol and or methanol, respectively.

Sample

Table 4 lists the samples for low-temperature and high-temperature tests.

Table 4. Samples for VT Calibration Curve

Temperature Range (°C)	*Sample Tube (mm)*	*Test Sample*	*Sample Part Number*
–50 to +25 (Low)	5	100% methanol (reagent grade)	00-968120-80
+25 to +100 (High)	5	100% ethylene glycol (reagent grade)	00-968120-79

Probe and Hardware Requirements

The variable temperature accessory and a VT probe are required.

VT tests should be run using a 5-mm probe that is capable of ^{1}H direct observe over the temperature range of -150°C to +200°C. For probes that have a more limited temperature range (particularly PFG probes), run the test at two or three temperatures that fall within the VT range of the probe. These tests can also be run using the ^{1}H decoupling coil of the 5-mm broadband probe as ^{1}H direct observe.

Test Procedure for High-Temperature Calibrations

1. Check that the ^{1}H quarter-wavelength cable is installed and the probe is tuned. Use the following sample e for shimming up the probe prior to the VT calibrations:
   - Doped 2-Hz H_2O/D_2O (0.1 mg/ml $GdCl_3$ in 1% H_2O in D_2O), Part No. 01-901855-01 for 5-mm samples.
2. Enter **`rtp('/vnmr/tests/shmd2o')`** to retrieve the test parameter set to the current experiment. Acquire a normal spectrum and shim the water signal to about 3 to 4 Hz linewidth at 50%.

3. Replace the 99.8% D_2O sample with the 100% ethylene glycol sample (Part No. 00-968120-79). Set the following parameters: **pw=2 gain=5** (or some value that doesn't overload the receiver) **sw=10000 at=2 nt=1 in='n'**.

   In the **acqi** window, set the lock to Off. The test is run unlocked, because the sample has no deuterated solvent to lock on. Enter **su** and check the probe tuning for the ethylene glycol sample. Enter **ga** to acquire the spectrum. Place the cursor between the two peaks and enter **movetof** to move the transmitter offset.

4. Click the mouse on the **Box** menu button to call up right and left cursors. Position the right and left cursors on the right and left peaks. Enter **tempcal('glycol')**.

5. Record the temperature reading from the VT controller (displayed on the remote status module or on the front face of the VT controller) and the computer-calculated temperature based on the chemical shift frequencies of the two peaks.

CAUTION: **Extreme temperatures can damage the probe. The high and low temperatues must be within the specified range of the probe.**

6. Enter **temp=50 su** to change the temperature to 50°C. Allow the sample to stabilize at 50° C for at least 10 minutes after the VT controller has reached the final temperature and regulated. Enter **ga** to acquire a spectrum. Repeat step 3 above and record the two temperatures.

7. Make sure that the VT gas flow and cooling air flow levels are between 9.5 to 10 LPM and gas flow to the probe is not restricted in any way. Enter **temp=100 su** to change the temperature to 100°C.

8. Allow the sample to stabilize at 100°C for at least 10 minutes after the VT controller has reached the final temperature and regulated. Enter **ga** to acquire a spectrum. Repeat step 3 above and record the two temperatures.

Test Procedure for Low-Temperature Calibrations

CAUTION: **For low-temperature calibrations, fill the VT dewar with liquid nitrogen. If a chemical mixture is used instead of liquid nitrogen for low-temperature calibrations, choose the chemical slurry carefully. A mixture of crushed dry ice and acetone is not recommended, because it will dissolve the polystyrene VT dewar.**

1. Check that the ^1H quarter-wavelength cable is installed and the probe is tuned. Use the following sample for shimming up the probe prior to the VT calibrations:
   - Doped 2-Hz H_2O/D_2O (0.1 mg/ml $GdCl_3$ in 1% H_2O in D_2O), Part No. 01-901855-01 for 5-mm samples.

2. Enter **rtp('/vnmr/tests/shmd2o')** to retrieve the test parameter set to the current experiment. Acquire a normal spectrum and shim the water signal to about 3 to 4 Hz linewidth at 50%.

3. Replace the 99.8% D_2O sample with the 100% methanol sample (Part No. 00-968120-80). Set the following parameters: **pw=2 gain=5** (or some value that doesn't overload the receiver) **sw=10000 at=2 nt=1 in='n'**.

   In the acqi window, set the lock to Off. The test is run unlocked because the sample lacks deuterated solvent to lock on.

Enter **su** and check the probe tuning for the methanol sample. Enter **ga** to acquire the spectrum. Place the cursor between the two peaks and enter **movetof** to move the transmitter offset.

4. Click on the **Box** menu button to call up right and left cursors. Position the right and left cursors on the right and left peaks, and enter **tempcal('methanol')**.
5. Record the temperature reading from the VT controller (displayed on the remote status module or on the front face of the VT controller). Record also the computer-calculated temperature based on the chemical shift frequencies of the two peaks. If low-temperature calibrations are performed immediately following high-temperature calibrations, allow the probe to cool to room temperature before continuing with the rest of the procedure.
6. Enter **temp=-20 su** to change the temperature to –20° C. Allow the sample to stabilize at –20° C for at least 10 minutes after the VT controller has reached the final temperature and regulated. Enter **ga** to acquire a spectrum. Repeat step 3 above and record the two temperatures.
7. Enter **temp=-80 su** to change the temperature to –80° C. Allow the sample to stabilize at –80° C for at least 10 minutes after the VT controller has reached the final temperature and regulated. Enter **ga** to acquire a spectrum. Repeat step 3 above and record the two temperatures.
8. After finishing the low-temperature test, enter **temp='n' su** to turn off the temperature regulation. While keeping the dry nitrogen gas flowing to the probe and upper barrel, remove the polystyrene VT dewar containing liquid nitrogen. The flow of dry nitrogen gas to the probe will prevent condensation inside the probe. Allow the dry nitrogen gas to flow through the probe and upper barrel for at least 15 minutes while the probe warms up to room temperature.
9. Plot a graph of the VT controller reading (horizontal axis) as compared with the calculated VT reading from the chemical-shift differences between the two peaks (vertical axis). Draw a straight line through the points.

Stability Calibration for High-Stability VT Accessory

This test is for high-stability VT units only (Part No. 00-992953-00). It demonstrates that the VT unit can hold the temperature within ± 0.1°C. The test requires preconditioning of the laboratory air and applies restrictions on the room temperature fluctuations.

Sample

Doped 2-Hz H_2O/D_2O (0.1 mg/ml $GdCl_3$ in 1% H_2O in D_2O), Part No. 01-901855-01 for 5-mm samples.

Alternatively, the customer can use a 10-mM DSS in D_2O (sample volume of 0.6 ml in a 5-mm NMR tube) DSS= 3-(trimethylsilyl)-1-propane sulfonic acid. The customer must make this sample using DSS and deuterium oxide (99.8 or 99.9 atom%D). Upon request, Varian can make this sample if DSS is not available at the customer site.

Probe and Hardware Requirements

High-stability variable temperature accessory and a 5-mm probe capable of ^1H direct observe are required.

Test Procedure

1. Enter **`rtp('/vnmr/tests/shmd20')`** to retrieve the test parameter set to the current experiment.
2. Enter **`temp=40 at=10 sw=10000`**. Set **`pw`** to the ^{1}H 90° pulse width for the probe, and then enter **`spin='n' su`**.

   Allow the VT controller to regulate to 40° C, which should be about 10° higher than the room temperature. Check that the ^{1}H quarter-wavelength cable is installed (200-, 300-, and 400-MHz systems) and the probe is tuned.
3. Acquire a normal spectrum by entering **`ga`**. Move the cursor to the DSS signal (right-most peak). Enter **`movetof sw=1000 at=10`**.
4. Acquire a normal spectrum and shim the DSS signal to about 0.6 Hz or less linewidth at 50%. The sample of DSS in D_2O should equilibrate at 40° C for at least 2 hours before the next step.
5. Enter **`in='n' spin='n' nt=1`** and **`array('d1',73,600,0) d1[1]=0`**.

   This sets up an array of `d1` values with the first spectrum to be collected at time 0 minutes, and subsequent spectra to be collected at 10 minute intervals for up to 12 hours. Enter **`ga`** to acquire the spectra. The test takes 10 to 11 hours to complete.
6. After the spectra are acquired, phase the first spectrum by entering **`ds(1)`** to display the first spectrum of the array, and by entering **`aph0`** to apply a first-order phase correction to the spectrum.
7. Enter **`ai`** to scale all of the spectra to the same vertical scale, and enter **`dssa`** to display the arrayed spectra stacked vertically.
8. Measure the difference between the left-most peak and the right-most peak in Hz.

Homospoil Demonstration

Sample

Doped 2-Hz H_2O/D_2O (0.1 mg/ml $GdCl_3$ in 1% H_2O in D_2O), Part No. 01-901855-01 for 5-mm samples.

Probe and Hardware Requirements

A 5-mm probe capable of ^{1}H direct observe is recommended.

Test Procedure—Part 1

1. Enter **`rtp('/vnmr/tests/shmd20')`** to retrieve the test parameter set to the current experiment. Enter **`solvent='d2o' pw=0 hst=0.005 sw=6000 d1=60 d2=0.055`**; then set **`p1`** to the ^{1}H 90° pulse width value and enter **`su`**. Check that the appropriate quarter-wavelength cable for ^{1}H is installed (200-, 300-, and 400-MHz systems) and the probe is tuned.
2. Enter **`ga`** to acquire a normal spectrum. Shim the water signal to about 3 Hz or less linewidth at 50%. Set **`tof`** to be about 100 Hz off resonance from the water peak.
3. Array the homospoil parameter by entering **`hs='nn','ny'`**. Enter **`ga`** to acquire the arrayed spectra. Enter **`ds(1)`** to display the first spectrum. Phase the spectrum by entering **`aph0`**. Display both spectra by entering **`dssh`**.

The second spectrum should be considerable smaller than the first spectrum.

4. Measure the amplitude of the first spectrum by moving the cursor on top of the peak and entering **nl**.

   The height of the peak (in mm) and the frequency are displayed in the status window. Record the height of the peak for the first spectrum. Measure the amplitude of the second spectrum by displaying the second spectrum using **ds(2)**. Make sure the first and second spectra are displayed using the same values of **vs** (vertical scale). Move the cursor on top of the peak and enter **nl**. Record the height of the peak for the second spectrum.

5. Calculate the residual signal amplitude of the second spectrum as follows:.

$$\frac{h_2 \times 100}{h_1} = \textit{Percentage of residual signal amplitude}$$

   Where h_2 is the height of the second spectrum and h_1 is the height of the first spectrum.

Test Procedure—Part 2

1. Enter **rtp('/vnmr/tests/shmd20') p1=0 hst=0.005 sw=6000 d1=60 d2=0.055**; then set **pw** to the ^{1}H 90° pulse width value and enter **su**. Check that the appropriate quarter-wavelength cable for ^{1}H is installed (200-, 300-, and 400-MHz systems) and the probe is tuned.

2. Enter **ga** to acquire a normal spectrum. Shim the water signal to about 3 Hz or less linewidth at 50%. Set the **tof** to be about 100 Hz off resonance from the water peak.

3. Array the homospoil parameter by entering **hs='nn','ny'**. Enter **ga** to acquire the arrayed spectra. Enter **ds(1)** to display the first spectrum. Phase the spectrum by entering **aph0**. Display both spectra by entering **dssh**. The second spectrum should be considerably smaller than the first spectrum.

4. Measure the amplitude of the first spectrum by moving the cursor on top of the peak and entering **nl**. The height of the peak (in mm) and the frequency are displayed in the status window. Record the height of the peak for the first spectrum. Measure the amplitude of the second spectrum by displaying the second spectrum using **ds(2)**. Make sure the first and second spectra are displayed using the same values of **vs** (vertical scale). Move the cursor on top of the peak and enter **nl**. Record the height of the peak for the second spectrum.

5. Calculate the residual signal amplitude of the second spectrum as follows:.

$$\frac{h_2 \times 100}{h_1} = \textit{Percentage of residual signal amplitude}$$

   Where h_2 is the height of the second spectrum and h_1 is the height of the first spectrum.

Sucrose Anomeric ^{1}H Signal-to-Noise Ratio

This test measures signal-to-noise ratio for anomeric proton of 1.46 mM sucrose in D_2O (with and without 250 mM NaCl) for 500-, 600-, 750-, and 800-MHz systems.

Samples

The samples are the following:

- 300 µg sucrose in 0.6 mL of D_2O
- 300 µg sucrose, 250 mM NaCl in 0.6 mL of D_2O
- 0.6 mL sample volume in a standard 5-mm NMR sample tube (Wilmad 535-PP)

These samples can be made up by the customer or specially prepared samples from Varian can be sent for the test.

Probe and Hardware Requirements

This test requires 5-mm triple-resonance, indirect detection, and proton probes (probes capable of ^1H direct observe). The tests are designed to be performed at a power level giving rise to a 90° pulse width less than or equal to 10 µs (for the no-salt sample).

This test requires excellent B_0 homogeneity. As a prerequisite, the probe should be shimmed well enough to meet ^1H non-spin lineshape specifications. The sucrose ^1H signal-to-noise test is performed with the sample not spinning.

Test Procedure

1. Insert sample A into the magnet, tune the probe, and adjust the homogeneity using the lock signal.
2. Enter **`rtp('/vnmr/tests/shmd2o')`** and **`presat`** to retrieve the test parameter set to the current experiment.
3. Set **`nt=1 spin='n' sw=6000 at=5.0 gain=40 pw=10`** (^1H 90° pulse width) **`tpwr=57`** (or whatever `tpwr` was used to give a ^1H 90° pulse width of 10 µs or less) **`dm='nnn' satpwr=4 satfrq=tof presat=2.0 tnsat='y' composit='n' lb=1.0`**.
4. Enter **`ga`** to acquire the spectrum.

   If the receiver or the ADC overloads, reduce `gain` and reacquire the spectrum.
5. Phase the spectrum (using manual phasing), and place the cursor on the water signal.
6. Enter **`nl rl(4.8p) movetof satfrq=tof`** and reaquire the spectrum. Then adjust `gain` to the highest level without ADC overload.

   The sucrose anomeric proton peak will be at 5.3 ppm. Determine the ^1H 90° pulse width for the sample by arraying `pw` to determine the optimum `pw`.
7. Expand the spectral region between 5 to 7 ppm. The ^1H signal-to-noise of the anomeric proton of sucrose is measured using a 200-Hz noise region between 5.4 to 7.0 ppm.
8. Write the results on the forms provided in “Consoles and Magnets Custom Specifications Form,” page 81.
9. Insert sample B into the probe, tune the probe, and adjust the homogeneity using the lock signal.
10. Determine the ^1H 90° pulse width for the sample by arraying `pw`. Use the same `tpwr` value as used for the no-salt test.
11. Repeat the procedure for sample C.

The ^{1}H 90° pulse width for sample B is increased 20% to 25% over the sucrose with no salt sample.

12. Write the results for each probe in the forms provided in "Consoles and Magnets Custom Specifications Form," page 81.

Aqueous Phenylalanine Water Suppression

Sample

1 mM phenylalanine, 10-mM NaOAc, 10-mM TSP, 90% H_2O/10% D_2O (600 µl sample volume, Wilmad 535-PP) for 500-, 600-, 750-, and 800-MHz systems.

This sample can be made up by the customer can be specially prepared by Varian and sent for the test.

Probe and Hardware Requirements

Water suppression tests should be run using a 5-mm probe capable of ^{1}H direct observe. The tests are designed to be performed at a power level giving rise to a 10 µs 90° pulse width.

This test requires excellent B_0 homogeneity. As a prerequisite, the probe should be shimmed well enough to meet ^{1}H nonspin lineshape specifications.

Test

Use the following procedure:

1. Check that the probe is tuned and the 90° pulse width determined for a doped 2-Hz D_2O sample.

   The 90° pulse width determined from the doped D_2O sample (for example, 10 µs at `tpwr=58`) is used for the `pw` value for the water suppression test.

2. Obtain the correct frequency for the presaturation of the water peak as follows:

   a. Enter **`rtp('/vnmr/tests/shmd2o')`** to retrieve the test parameter set into the current experiment.

   b. Enter **`nt=4 sw=7500 at=0.8 tpwr=10 gain=20`**. Change **`pw`** to the 90° value and enter **`ga`** to acquire the spectrum.

      If the receiver or the ADC overloads, reduce `gain` and reacquire the spectrum.

   c. Phase the spectrum (using manual phasing), and place the cursor near the water signal. Enter **`nl rl(4.8p) movetof dof=tof`**.

   d. Enter **`presat d1=0.01 ss=2 nt=4 gain=20 lb=1 satfrq=tof presat=2 satpwr=4`** (or the `tpwr`-type power level provides a 50 Hz field on the transmitter, see step 3 for the calibration procedure) **`tpwr=63 pw=8`** (or whatever is the `tpwr` and `pw` values meet the ^{1}H 90° pulse width calibration specification for the probe).

   e. Enter **`ga`** to acquire the spectrum.

      The spectrum should show that the water peak is suppressed. The water peak should be roughly the same amplitude or smaller than the acetate (about 2 ppm) and the TSP (at 0 ppm) peaks.

If the water peak is not suppressed, the `satpwr` level may need to be calibrated to give a 50-Hz γH_2 field or the `tof` may need to be moved so that it is directly on the water peak.

3. The γH_2 field calibration is made by calibrating the 90° pulse. If the water peak is used to calibrate the 90° pulse, the water signal would overload the system. A better way is to apply a pulse to the TSP (0 ppm) line. Calibrate the γH_2 field as follows:

   a. Place the cursor on the TSP resonance (the line furthest to the right as you look at the spectrum from the previous steps).

   b. Enter **`nl movetof`**.

   c. Set **`d1=10 nt=1 pw=5000`**.

      The relationship between the desired γH_2 field and `pw` is `pw`= 1/(4*γH_2 field). For a 50-Hz γH_2 field, `pw=5000`.

   d. Array **`tpwr`** over a range of values that span the desired power level (a suggested array is `tpwr=4,6,8,10,12,14,16,18,20`).

      Enter **`wexp='wft dssh' au`**.

   e. Select the **`tpwr`** value that gives the maximum signal amplitude from the previous step.

   f. Enter **`setlimit('pw',50000,0,0.025)`** to allow the software limit for `pw` to exceed 8192.

   g. Array **`pw`** around the 360° pulse (4 * 90° pulse). A suggested array is `pw=16000,18000,20000, 22000,24000,26000`.

   h. Select the γH_2 value that gives the best null signal. The exact γH_2 field is 1/360° pulse width. Substitute the `tpwr` value that gives the desired γH_2 field for the value of `satpwr` in step 2d.

4. With the `tpwr` that gives the desired γH_2 field substituted for the `satpwr`, move the cursor near the water peak and enter **`nl rl(4.8p) movetof dof=tof`**. Enter **`ga`** to acquire the spectrum. Measure the width of the suppressed water peak at the height of the phenylalanine multiplet near 4 ppm.

5. Write the results for each probe on the forms provided in “Consoles and Magnets Custom Specifications Form,” page 81.

Chapter 3. Consoles and Magnets Specifications

Sections in this chapter:

- 3.1 “Specifications for AutoTest” this page
- 3.2 “Specifications for Manual Console Tests” page 60
- 3.3 “Contracted Custom Console Specifications” page 67

Each section contains the published specifications for UNITY*INOVA* consoles and magnets. Chapter 2 contains the corresponding acceptance test procedures.

3.1 Specifications for AutoTest

All AutoTest data is stored, and plots and statistical analyses are provided as part of the acceptance testing. Plots and statistical analyses are made concurrently with acquisition.

Sample

The AutoTest sample is 0.1% ^{13}C enriched methanol in 1% H_2O/99% D_2O.

The sample is doped with gadolinium chloride at a concentration of 0.30 mg/ml, which produces a ^{1}H T_1 relaxation time of about 50 to 75 ms. The resulting linewidth is between 6 and 10 Hz.

Hardware Requirements

The rf system (transmitters, linear modulators, rf attenuators, amplifiers, receivers, and probes) must be in the standard configuration when AutoTest is run. If the system configuration has been changed, it must be returned to the standard configuration before running AutoTest for acceptance testing.

Basic Specifications

Table 5 lists the specifications and demonstrations generated by AutoTest. Gradient pulses are rectangular except where noted. Oversampling and/or digital filtering is disabled except when doing the frequency-shifted image and receiver gain tests.

Note 1 – Specifications for 90° pulse stability and 30° amplitude stability are given in “% instability,” which is 100% minus the standard deviation of the intensity (in %).

Note 2 – Phase cycle cancellation and gradient phase cycle cancellation specifications depend on floor vibrations not exceeding the levels specified in the UNITY*INOVA Installation Planning* manual.

For attenuator linearity tests, the standard deviation (std. dev.) value is provided as a reference but is not a specification for the test.

Table 5. AutoTest Specifications and Demonstrations

Test	*AutoTest Specification*
RF homogeneity test: ^{1}H	^{1}H rf homogeneity spec. for probe
RF homogeneity test: ^{13}C	^{13}C rf homogeneity spec. for probe
Water resonance frequency	value reported
T_1	value reported
^{1}H PW90 determination for channels 1 and 2	^{1}H PW90 spec. for probe
^{13}C PW90 determination	^{13}C PW90 spec. for probe
High-band amplifier compression	measure
Low-band amplifier compression	measure
Temperature rise in decoupler heating test	demonstration
AutoGain test for 90° pulse	demonstration
Signal-to-noise (normal and oversampled)	demonstration
Folded noise reduction with large SW	demonstration
Average signal-to-noise (20 trials)	demonstration
^{13}C phase modulation decoupling profile (using phase modulator)	
GARP-1 modulation	demonstration
WALTZ-16 modulation	demonstration
XY-32 modulation	demonstration
MLEV-16 modulation	demonstration
^{13}C adiabatic decoupling profiles (using waveform generator)	
STUD modulation	demonstration
WURST modulation	demonstration
Receiver gain tests (normal and oversampled)	demonstration
Small-angle phase test (channels 1 and 2)	demonstration
90° pulse stability (channels 1 and 2) (see Note 1)	≥ 99.9%
Phase cycle cancellation – 2 scans (see Note 2)	measure
Phase cycle cancellation – 4 scans (see Note 2)	≤ 0.25%
Quadrature images and spurious signals – 1 scan	≤ 0.4%
Quadrature images and spurious signals – 4 scans	≤ 0.04%
Frequency-shifted quadrature image – 1 scan	0.05%
Frequency-shifted quadrature image – 4 scans	0.05%
30° amplitude stability (channels 1 and 2) (see Note 1)	≥ 99.9%
Pulse turnon time for channel 1	≤ 0.05 µs
Pulse turnon time for channel 2	≤ 0.05 µs
Attenuator linearity tests (channels 1 and 2; full power)	
400 MHz systems and above	corr. coef. = 0.95 ±0.03, std. dev. = 0.007*
200 and 300 MHz systems	corr. coef. = 0.92 ±0.05, std. dev. = 0.01*
Attenuator linearity test (channels 1 and 2; reduced power)	
400 MHz systems and above	corr. coef. = 0.98 ±0.03, std. dev. = 0.007*
200 and 300 MHz systems	corr. coef. = 0.92 ±0.05, std. dev. = 0.02*

Table 5. AutoTest Specifications and Demonstrations *(continued)*

Test	*AutoTest Specification*
Modulator linearity (channel 1; `tpwr` = 40)	std. dev. ≤0.25
Modulator linearity (channel 2; `tpwr` = 40)	std. dev. ≤0.25
Modulator linearity (channel 1; `tpwr` = –16)	std. dev. ≤ 0.50
Modulator linearity (channel 2; `dpwr` = –16)	std. dev. ≤ 0.50
13° phase error (channels 1 and 2)	≤ 0.09°
Gradient level for 10 G/cm along Z	value reported
Gradient level for 10 G/cm along X	value reported
Gradient level for 10 G/cm along Y	value reported
Z-gradient echo stability (30 G/cm)	≥ 99.9%
X-gradient echo stability (10 G/cm)	≥ 99.9%
Y-gradient echo stability (10 G/cm	≥ 99.9%
Z-gradient echo stability (10 G/cm)	≥ 99.9%
CPMG T_2 calculation (gradients on, mismatched, and off)	demonstration
Z-gradient recovery stability (10 G/cm)	≥ 99.9%
X-gradient recovery stability (10 G/cm)	≥ 99.9%
Y-gradient recovery stability (10 G/cm)	≥ 99.9%
Z-axis signal recovery (rect)	≤ 50 µs
Z-axis signal recovery (sine)	≤ 50 µs
X-axis signal recovery (rect)	≤ 30 µs
X-axis signal recovery (sine)	≤ 30 µs
Y-axis signal recovery (rect)	≤ 30 µs
Y-axis signal recovery (sine)	≤ 30 µs
Gradient phase cycle cancellation – 4 scans (see note 2)	≤ 0.5%
Gaussian 90° stability (channels 1 and 2)	≥ 99.9%
Gaussian 13° phase error (channels 1 and 2)	≤ 0.09°
GaussianSLP 13° phase error (channels 1 and 2)	≤ 0.09°
Temperature rise in spinlock test	demonstration
Lock power test: corr. coef.	≥ 0.85
Lock gain test: corr. coef.	≥ 0.9

* standard deviation (std. dev.) provided as reference, but is not a specification for the test.

3.2 Specifications for Manual Console Tests

This section contains the following specifications:

"Lock Frequency Stability," this page
"Homonuclear Decoupling," this page
"Variable Temperature Operation," page 63
"Magnet Drift Specifications," page 64
"WALTZ 1H Decoupling Using Preprogrammed Phase Modulation," page 65
"WALTZ 1H Decoupling Using High-Performance Waveform Generators" page 66

Lock Frequency Stability

Sample

99% D_2O/1% H_2O in a 5-mm sample tube (0.7 µl volume)

Probe and Hardware Requirements

A 5-mm probe capable of ^1H direct observe is recommended. This test also requires a Lock Module on the spectrometer.

Basic Specifications

The lock is stable to within 0.01 Hz, as judged by the phase response of a Hahn echo in 99% D_2O/ 1% H_2O with 500 ms echo interval (0.1 Hz = 36° zero-order phase change). The maximum phase variation in the twenty experiments must not exceed ±3.6°.

Homonuclear Decoupling

Sample

0.1% ethylbenzene, 0.01% TMS, 99.89% deuterochloroform ($CDCl_3$)

Part No.	*Sample Tube*
00-968120-70	5 mm
00-968123-70	10 mm

Probe and Hardware Requirements

A 5-mm probe capable of ^1H direct observe is recommended.

Basic Specifications

The quartet shows a single peak with no remaining evidence of splitting.

Variable Temperature Operation

For basic variable temperature (VT) accessories (Varian Part No. 00-992957-00), demonstrate that the VT unit and probe go to the desired temperature as registered on the window of the VT controller. If the system is equipped with a VT unit, the system user should read through the VT operation instructions before the demonstration.

Dry nitrogen is required as the VT gas if the requested temperature is over 100°C or below 10°C. Otherwise, air can be used. For temperatures below –40°C, dry nitrogen gas is recommended for cooling the bearing, spinner, and decoupler. This prevents moisture condensation in the probe and spinner housing.

CAUTION: **The use of air as the VT gas for temperatures above 100°C is not recommended. Such use destructively oxidizes the heater element and the thermocouple.**

CAUTION: **Extreme temperatures can damage the probe. The high and low temperature must be within the specified range of the probe.**

Demonstration Limitations

If dry nitrogen gas and liquid nitrogen are not available at the time of installation, the range of VT demonstration is limited to temperatures between 30°C and 100°C.

Sample

Insert an empty sample tube with spinner into the probe.

Probe and Hardware Requirements

Any variable temperature probe is used.

Basic Specifications

The specifications for variable temperature ranges are listed with each probe.

Magnet Drift Specifications

Sample

For 200- and 300-MHz systems, use ^{1}H lineshape sample 20% $CHCl_3$ in 80% deuteroacetone $(CD_3)_2CO$, Part No. 00-968120-76.

For 400-, 500-, 600-, and 750-MHz systems, use 1% $CHCl_3$ in 99% deuteroacetone $(CD_3)_2CO$, Part No. 00-968120-89

Probe and Hardware Requirements

A 5-mm probe capable of ^{1}H direct observe is recommended.

Basic Specifications

Table 6 lists the drift specifications for magnets. Specifications for nominal field decay rate are less than or equal to the values listed in the table.

Table 6. Magnet Drift Specifications

System (MHz/mm)	*Field Strength (T)*	*Nominal Field Decay Rate (Hz/hr)*
200/54, 200/89	4.70	2
300/54, 300/89	7.05	3
400/54	9.40	8
400/89	9.40	10
500/51	11.75	10
600/51	14.10	10
750/51	17.60	15

WALTZ ^{1}H Decoupling Using Preprogrammed Phase Modulation

This test is performed using the phase modulator and not the waveform generators.

Sample

Doped 40% p-dioxane in benzene-d_6.

Part No.	*Sample Tube*
00-968120-91	5-mm
00-968123-91	10-mm

Probe and Hardware Requirements

A 5-mm or 10-mm broadband observe, ^{1}H decouple probe is recommended (i.e. 5-mm or 10-mm broadband probes, 5-mm switchable probes, or 4-nucleus probes). This test requires the results from the γH_2 calibration that gives a `dpwr` of 1 watt at the probe.

Basic Specifications

All of the spectra in the array show a single peak. With the exception of the first and last spectrum, the remaining spectra do not vary in amplitude by more than 10% of the average amplitude.

WALTZ ^{1}H Decoupling Using High-Performance Waveform Generators

This test is performed by generating shaped pulses using the waveform generators.

Sample

Doped 40% p-dioxane in benzene-d_6.

Part No.	*Sample Tube*
00-968120-91	5-mm
00-968123-91	10-mm

Probe and Hardware Requirements

A 5-mm or 10-mm broadband observe, ^{1}H decouple probe is recommended (i.e. 5 mm or 10 mm broadband probes, 5 mm switchable probes, or 4-nucleus probes). This test requires the presence of waveform generator boards. This test also requires the results from the γH_2 calibration that gives a `dpwr` of 1 watt at the probe.

Basic Specifications

All of the spectra in the array show a single peak. With the exception of the first and last spectrum, the remaining spectra do not vary in amplitude by more than 10% of the average amplitude.

WALTZ decoupling using programmable decoupling should be comparable to the non-programmable (hardware) WALTZ. Compare the results obtained in this test to those obtained from the test “WALTZ 1H Decoupling Using Preprogrammed Phase Modulation,” page 65.

Note that files with other modes of decoupling can be found in the `/vnmr/shapelib` directory as `pulseshape.DEC`.

3.3 Contracted Custom Console Specifications

This section contains the following specifications:

"Temperature Accuracy for VT Accessories," this page
"Stability Calibration for High-Stability VT Accessory," page 69
"Homospoil Demonstration," page 70

Before testing to specifications in this section, refer to"Consoles and Magnets Custom Specifications Form," page 81, for any custom acceptance test specifications.

Note: Specifications on a Custom Specifications Form are done only by prior contract agreement and supersede the specifications shown in this manual.

Temperature Accuracy for VT Accessories

Sample

For high-temperature work—100% ethylene glycol (reagent grade)
Part No. 00-968120-79 for 5-mm sample tubes

For low-temperature work—100% methanol (reagent grade)
Part No. 00-968120-80 for 5-mm sample tubes

Table 7 lists which sample to use with a temperature range for VT calibration curve.

Table 7. Samples for VT Calibration Curve

Temperature Range (°C)	*Sample Tube (mm)*	*Test Sample*	*Sample Part No.*
–50 to +25 (Low)	5	methanol	00-968120-80
+25 to +100 (High)	5	ethylene glycol	00-968120-79

Probe and Hardware Requirements

A VT Unit and a VT probe are required fore these tests.

For best results, run the VT tests using a 5-mm probe that is capable of ^{1}H direct observe over the temperature range of –150°C to +200°C. For probes that have a more limited temperature range (particularly PFG probes), run the test at two or three temperatures that fall within the VT range of the probe. These tests can also be run using the ^{1}H decoupling coil of the 5-mm broadband probe as ^{1}H direct observe.

CAUTION: **Before performing variable temperature calibrations, check that the VT unit is correctly installed according to the instructions in the installation manual. Dry nitrogen gas should be used for the gas flow to the probe and upper barrel for both the high- and low-temperature calibrations.**

CAUTION: **For the low-temperature calibrations, fill the VT dewar with liquid nitrogen. If a chemical mixture is used instead of liquid nitrogen for the low-temperature calibrations, be careful about the choice of the chemical slurry. A mixture of crushed dry ice and acetone is not recommended, because it dissolves the polystyrene VT dewar.**

Basic Custom Specifications

The temperature reading displayed on the VT unit display panel should be within ±1° C of the actual temperature reading, as measured from the chemical shift.

Stability Calibration for High-Stability VT Accessory

Sample

Doped 2-Hz H_2O/D_2O (0.1 mg/ml $GdCl_3$ in 1% H_2O in D_2O), Part No. 01-901855-01 for 5-mm samples.

Alternatively, the customer can request 10-mM DSS in D_2O (sample volume of 0.6 ml in a 5-mm NMR tube) DSS= 3-(trimethylsilyl)-1-propane sulfonic acid. The customer must make this sample using DSS and deuterium oxide (99.8 or 99.9 atom%D). Upon request, Varian can make the sample if DSS is not available at the customer site.

Probe and Hardware Requirements

The high-stability VT Accessory and a 5-mm probe capable of ^1H direct observe are required. The high-stability VT tests have the following customer site prerequisites:

1. Room temperature regulation to within ±1°C, as described in the manual UNITY*INOVA Installation Planning*.
2. Dry nitrogen for the air supply for temperatures below 10°C, per the guidelines in the manual UNITY*INOVA Installation Planning*.
3. A preconditioned VT air supply, with the VT air cooled to ≤ 10°C below the desired sample temperature.
4. The air flow to the probe for VT regulation in accord with the Varian probe manuals.

Specifications for High-Stability VT Units

The high-stability VT accessory holds the set temperature to within ±0.1°C. (±0.1°C = 0.001 ppm: 600 MHz, ±0.6 Hz; 500 MHz, ±0.5 Hz; 400 MHz, ±0.4 Hz)

Homospoil Demonstration

Sample

Doped 2-Hz H_2O/D_2O (0.1 mg/ml $GdCl_3$ in 1% H_2O in D_2O), Part No. 01-901855-01 for 5-mm samples.

Probe and Hardware Requirements

Use a 5-mm probe capable of ^1H direct observe.

Specifications

Part 1: The residual signal amplitude of the second spectrum is less than 5% of the signal amplitude of the first spectrum.

Part 2: The residual signal amplitude of the second spectrum is greater than or equal to 95% of the signal amplitude of the first spectrum.

Chapter 4. Acceptance Test Results

Sections in this chapter:

- 4.1 “Computer Audit” page 73
- 4.2 “System Installation Checklist” page 75
- 4.3 “Supercon Shim Values” page 77
- 4.4 “Console and Magnet Test Results” page 79
- 4.5 “Consoles and Magnets Custom Specifications Form” page 81

This chapter contains forms for recording system information and acceptance test results.

Notes:

4.1 Computer Audit

Information about your site (please print):

Company/University _____

Address _____

_____

Principal User _____

Phone _____ Spectrometer type _____

Fax _____ Console S/N _____

Sales Order No. _____ Delivery (month/day) _____

Information on each computer (additional forms are on the back of this page). Include computers directly attached to the spectrometer, computers (networked or non-networked, on-site or off-site) used to process NMR data using Varian's VNMR software, and computers (on-site and off-site) used to process data collected on this spectrometer with software from other vendors.

Information on computer ____ **of** ____ **(e.g., 1 of 3)**

Manufacturer _____ Model no. _____

Computer S/N _____ Purchased from _____

Memory (Mbytes) _____ Screen size (in.) _____

Peripherals: Internal hard disk (Mbytes) _____

External hard disk (Mbytes) _____ Serial no. _____

Tape drive size _____ Serial no. _____

CD-ROM drive model _____ Serial no. _____

Printer model _____ Serial no. _____

Plotter model _____ Serial no. _____

Terminal model _____ Serial no. _____

Other peripheral _____ Serial no. _____

Computer function: NMR host _____

Workstation running VNMR _____ on-site or off-site

Workstation running other NMR software _____ on-site or off-site

Workstation running VNMR and other NMR software _____ on-site or off-site

VNMR version _____ **Operating system** _____

The above computer audit was performed during installation of the system.

_____

Varian Representative Date

I certify that the information on this form is accurate and that all computers to be used to run VNMR software (including variants VnmrS, VnmrX, VnmrI, VnmrSGI, and VnmrV), or to run other software to process data obtained on this spectrometer, have been included in the audit (including those previously registered as part of purchases of other Varian NMR spectrometers).

_____

Customer Representative Date

Use these forms for additional computers. If more forms are needed, copy this page. Attach all copies to the Computer Audit.

Information on computer ____ of ____ (e.g., 2 of 3)

Manufacturer _____ Model no. _____

Computer S/N _____ Purchased from _____

Memory (Mbytes) _____ Screen size (in.) _____

Peripherals: Internal hard disk (Mbytes) _____

External hard disk (Mbytes) _____ Serial no. _____

Tape drive size _____ Serial no. _____

CD-ROM drive model _____ Serial no. _____

Printer model _____ Serial no. _____

Plotter model _____ Serial no. _____

Terminal model _____ Serial no. _____

Other peripheral _____ Serial no. _____

Computer function: NMR host _____

Workstation running VNMR _____ on-site or off-site

Workstation running other NMR software _____ on-site or off-site

Workstation running VNMR and other NMR software _____ on-site or off-site

VNMR version _____ **Operating system** _____

Information on computer ____ of ____ (e.g., 3 of 3)

Manufacturer _____ Model no. _____

Computer S/N _____ Purchased from _____

Memory (Mbytes) _____ Screen size (in.) _____

Peripherals: Internal hard disk (Mbytes) _____

External hard disk (Mbytes) _____ Serial no. _____

Tape drive size _____ Serial no. _____

CD-ROM drive model _____ Serial no. _____

Printer model _____ Serial no. _____

Plotter model _____ Serial no. _____

Terminal model _____ Serial no. _____

Other peripheral _____ Serial no. _____

Computer function: NMR host _____

Workstation running VNMR _____ on-site or off-site

Workstation running other NMR software _____ on-site or off-site

Workstation running VNMR and other NMR software _____ on-site or off-site

VNMR version _____ **Operating system** _____

4.2 System Installation Checklist

Company/University _____

Address _____

_____

Principal User _____

Phone _____ Spectrometer type _____

Fax _____ Console S/N _____

Sales Order No. _____ Magnet S/N _____

Shipment Damage:

_____

_____

Preinstallation Preparation:

Line voltage measured (Vac): console _____ accessory _____

Line pressure: air _____ N_2 _____

Air conditioning: _____

Cryogens (liters): LHe _____ LN _____

Testing and Customer Familiarization:

1. Acceptance tests and computer audit
   - ___ Acceptance tests procedures finished
   - ___ Test results form completed and signed
   - ___ Computer audit completed and signed
2. System documentation review
   - ___ *Software Object Code License Agreement* (acceptance of product constitutes acceptance of object code license regardless of whether agreement is signed or not)
   - ___ Varian and OEM manuals
   - ___ Explanation of warranty and where to telephone for information
3. Magnet demonstration
   - ___ Posting requirements for magnetic field warning signs
   - ___ Warning signs posted
   - ___ Cryogenics handling and safety
   - ___ Magnet refilling
   - ___ Flowmeters
   - ___ Homogeneity disturbances
4. Console and probe demonstration

   CAUTION! To avoid possible preamplifier damage, make sure the probe is connected and tuned to resonance.
   - ___ Loading programs, operating the streaming tape unit
   - ___ Experiment setup
   - ___ Basic operation to obtain typical spectra
   - ___ Demonstration of broadband operation
   - ___ Demonstration of homonuclear and heteronuclear decoupling

Notes:

4.3 Supercon Shim Values

Fill in the following information:

Magnet Frequency and Serial Number:

Magnet Frequency _____

Serial Number _____

Measurement in:

Helipot _____

Amps _____

Measurement	1. Date:	2. Date:	3. Date:
Z0			
Z1			
Z2			
Z3			
Z4			
X			
Y			
ZX			
ZY			
XY			
X2–Y2			
Drift			
Spacers			
Main Field Current			
Customer Signature:			
Varian Representative Signature:			

Notes:

4.4 Console and Magnet Test Results

Fill in the following information.

Homonuclear Decoupling:

Lock Frequency Stability:

Basic Variable Temperature Operation:

Magnet Drift:

WALTZ ^{1}H Decoupling—Preprogrammed Phase Modulation:

WALTZ ^{1}H Decoupling—High-Performance RF Waveform Generator:

Varian Representative Date

Customer Representative Date

Notes

4.5 Consoles and Magnets Custom Specifications Form

Fill in the following information.

Custom Specification

Sample Requirements

Name of Procedure Required for Custom Specification:

If there is a test in this manual that can be modified for this custom specification, attach the necessary procedure to this form.

Numerics

180° pulse, 13
90 degree pulse width calibrations (PW90), 23
90° pulse value for pw, 13

A

acceptance tests documentation, 14
acceptance tests objectives, 11
automated console acceptance tests using shaped rf, 41
automated console tests, 21
automated decoupling performance tests, 23
automated tests with shaped rf, 23
automatic teller machine (ATM) cards caution, 9
AutoTest
  13C 90 degree Pulse Width Calibration, 31
  13C test descriptions, 30
  CPMG T2, 32
  creating probe-specific files, 40
  directory structure, 16
  experiment details, 24
  gradient tests descriptions, 31
  macros, 19
  other test descriptions, 33
  RF performance test descriptions (nonshaped channels 1 and 2), 24
  sample requirement, 38
  saving data and FID files from previous runs, 39
  standard tests, 21

B

basic system operation, 12
broadband operation, 12

C

cautions defined, 7
computer audit, 11
computer audit form, 73
console acceptance demonstration tests, 42
console acceptance tests, 37
console acceptance tests using AutoTest, 40
console demonstration, 12
console demonstration tests, 24
credit cards caution, 9
cryogenics handling procedures, 12

D

decoupling, 12
demonstrate console operation, 42
demonstration of system, 12
demonstration of the system, 12

E

ethylene glycol, 48
experiment setup, 12

F

flammable gases warning, 8
flowmeters, 12

G

gradient calibrations and performance tests, 23

H

H1sn test file, 43
Hahn echo, 43
helium contact with body, 8
helium gas flowmeters caution, 10
heteronuclear decoupling performance tests, 41
high-power amplifiers cautions, 10
high-stability VT units, 50
homogeneity disturbances, 12
homogeneity settings, 13
homonuclear decoupling test, 43
homospoil demonstration, 51

I

installation checklist, 12
installation engineer, duties, 11
installation of AutoTest, 37
installation planning guide, 13

L

lineshape measurements, 14
linewidth measurement, 14
liquid nitrogen, 44, 63
loading programs, 12
lock stability manual test procedures, 43

M

magnet acceptance tests, 37
magnet demonstration, 12
magnet quench warning, 8
magnet refilling, 12
magnetic media caution, 9
metal objects warning, 7
methanol, 48
modifying the instrument, 8

N

90° pulse, 13
nitrogen contact with body, 8
nitrogen gas, 44, 63
nitrogen gas flowmeters caution, 10

noise region, 14

O

objectives of acceptance tests, 11
OEM manuals, 12
180° pulse, 13

P

pacemaker warning, 7
parameters for tests, 13
phase stability, 29
preinstallation checklist, 75
probe demonstration, 12
probe performance tests, 41
prosthetic parts warning, 7
`H1sn` test file, 43
`pw` parameter, 13

Q

quarter-wavelength cable, 13

R

radio-frequency emission regulations, 10
relief valves warning, 9
removable quench tubes warning, 9
RF homogeneity tests, 23
`rtp` command, 13
`rts` command, 13
running AutoTest, 38

S

safety precautions, 7, 9
sensitivity tests, `pw` parameter, 13
set up of AutoTest, 38
shaped pulse test descriptions (channels 1 and 2), 29
shaped rf demonstrations, 24
shim parameters, 13
shipment damage, 75
signal-to-noise, 13
Software Object Code License Agreement, 12
solids high-power amplifiers caution, 10
spinning speed, 13
streaming magnetic tape unit, 12
`svs` command, 13
system demonstration, 12
system documentation review, 12
system installation checklist, 75

T

temperature accuracy for VT systems, 48
test conditions, 13
test parameters, 13
tests library, 13
tpwr value for AutoTest, 38
training seminars, 12

U

upper barrel warning, 8

V

variable temperature (VT) units, 44
Varian manuals, 12
vortexing, 13
VT controller, 44
VT experiment warning, 8

W

warnings defined, 7
warranty coverage, 12